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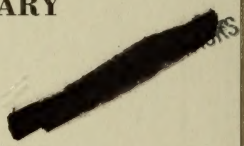

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INTERNATIONAL ASSOCIATION

FOR

TESTING MATERIALS

REPORT ON

PROBLEMS 1-37

620.6
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APR 25 1911
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On the basis of existing specifications to seek methods and means for the introduction of international specifications for testing and inspecting iron and steel of all kinds. (Proposed at the Zürich Congress 1895. Enlarged at the Budapest Congress 1901).

Report on problem 1. Presented by the Chairman of Commission 1, Dr. Ing. A. Rieppel, Nürnberg.

Chairman:

Rieppel A., Dr. Ing., k. Baurat, Direktor der „Vereinigte Maschinenfabrik Augsburg und Maschinenbaugesellschaft Nürnberg, A.-G.“, Äußere Cramer-Klettstraße 12.

Vice-Chairman:

Alpherts G., President of the Technical Bureau in the Dutch Colonial Ministry, Hague, Koninginnegracht 66.

Members:

Belgium: Verhoop W. H., Engineer to the Technical Bureau of the Dutch Colonial Ministry, Liège, Quai de Fragnée 10.

Denmark: Öllgaard F., Director of the Copenhagen Waterworks; Copenhagen V.

Germany: Martens A., Geheimer Regierungsrat, Professor, Dr. Ing., Direktor des königl. Materialprüfungsamtes; Groß-Lichterfelde-West.

England: Unwin W. C., Professor, F. R. S. M. I. C. E; 7 Palacegate Mansions; Kensington, London.

Wicksteed J. H. of Joshua Bukton a. Co. Ltd., Leeds.

France: Bécard A., ingénieur de la C^ie^ de l'ouest algérien; répétiteur et chef des essais de matériaux à l'école centrale; Paris, rue Miromesnil 2.

Baclé L., ingénieur; Paris, rue de Châteaudun 57.

Holland: Schroeder van der Kolk J., Ryksingenieur voor de Spoorwegen; s' Gravenhage.

Verhoop W. H., Engineer to the Technical Bureau of the Dutch Colonial Ministry; Liège, Quai de Fragnée 10.

Italy: Guidi C., ing.-prof. della R. Scuola d'applicazione per gli ingegneri di Torino, e direttore dell' annesso laboratorio per esperienze sui materiali da costruzione; Torino, Castello del Valentino.

Luxemburg: Mayrisch E., Direktor des Düdelinger Eisenhütten-Aktien-Vereines; Düdelingen.

Austria: Dormus A. von, Oberingenieur der K. F.-Nordbahn; Wien, II., Nordbahnstraße 50.

Russia: Belelubsky N., prof., directeur du laboratoire méc. à l'institute imp. des ing. des voies de comm., membre du conseil du ministère; St. Pétersbourg, 4 rue Serpouchowskaja.

Bogouslawsky N. B., ing. d'admin. centrale des chemins de fer de l'état russe; St. Pétersbourg, Razjezjaïa 15.

Chtchoukine N., prof., membre du cons. du min. des voies de comm.; St. Pétersbourg, Institute technologique.

Herberz B., directeur général de la société métallurgique de Jurjevsk; St. Pétersbourg, Wolkhowskoy per. 3.

Koslowsky B., ingénieur en chef du laboratoire mécanique; St. Pétersbourg, usines Poutilow.

Babouroff N., remplaçant de l'inspecteur en chef des usines métall.; St. Pétersbourg, Fontanka 117.

Sweden: Dillner G., Mining Engineer, Director of the R. Institute for Testing Materials of the Technical High School; Stockholm.

Switzerland: Schrafl Ant., Direktor der Gotthardbahn; Luzern.

Spain: Cano y de Leon M., ingénieur-civil; Madrid, rue Ayala 20.

Hungary: Tetmajer L. von (†), Director of the Iron Works Salgó-Tarján.

United States: Campbell H. H., Superintendent and General-Manager Pennsylvania Steel Company; Steelton, Penna.

Christie J, Mechan.-Engineer, Chief Mechanical Engineer, Am. Bridge Comp.; Pencoyd, Penna.

Carnegie Steel Company Ltd., John Mac Leod, Assistent to President; Pittsburg, Penna.

Franklin Institute, Dr. William H. Wahl, Secretary; 15 Sth. Seventh Street, Philadelphia, Penna.

Kreuzpointner P., Engineer of Tests, Penna. R. R. Altoona, Penna.

Webster W. R., Civil Engineer; 411—413 Walnut Street, Philadelphia, Penna.

Wood W., Cast Iron Pipe Manufacturer, R. D. Wood Comp; 400 Chestant Street, Philadelphia, Penna.

Moldenke Rich., Dr., Secretary of the Am. Foundrymens Association, P. O. Box, 432, New-York.

The last report was furnished in 1901 for the congress in Budapest and reference is accordingly made thereto in the following observation.

The comittee was unfortunately unable to report notable progress during this period. In most countries people concerned appear to have distinct objection to going into the matter. This is easily understood when one calls to mind that the contract specifications for iron and steel goods are in different countries so different that a universal system of contract specifications is hardly likely to be generally accepted. In many cases the solution of the problem is regarded as impossible, and it has accordingly not been approached with the desirable degree of enthusiasm. It is always to be assumed, however, that if general agreement regarding the conditions involved in specifications for iron and steel goods in the majority of countries were to be seriously sought for, an agreement of the majority of those interested could be arrived at.

The work has been undertaken in such a way in the various countries concerned that contract requirements may be capable of elaboration on general grounds. As soon as these separate proposals have been made, it is intended that the general committee shall examine them with a view to seeing how far they will enable a general proposal to be put forward on behalf of the Association.

The chairmen of the various committees are well aware that a recognition of proposals of this kind in the various countries concerned cannot be expected for some time to come. Nevertheless proposals which are agreed to by the Association, either unanimously or by a large majority, would probably receive serious consideration and act as guiding principles in a number of countries where consumers are to be found.

The present position of the work undertaken is as follows:

North-America: The American section of the International Association for Testing Materials used in Technology, placed its proposals for contract specifations before the Budapest Congress in 1901; they referred to

a) Specifications for Structural Steel for Bridges and Ships,
b) „ „ „ „ „ Buildings,
c) „ „ Open-Hearth Boiler-Plate and Rivet-Steel,
d) „ „ Steel-Rails,
e) „ „ „ Splice-Bars,
f) „ „ „ Tires,
h) „ „ „ Forgings,
i) „ „ „ Castings,
k) „ „ Wrought-Iron.

In this year the same committee put forward its proposals in regard to cast iron. This concluded the greater portion of the work they had untertaken.

France: In France there have been in existence for some ten years the very valuable collected work of Barba. In addition the committee collected all the contract specifications procurable up to 1901, and collated them, but they have not been put together in a very suitable form. Messrs. Bacle and Bacard are of the opinion that the work should be referred back again until the official committee which is dealing with the subject has finished its labours.

In October 1902 the reporters named write as follows:

»I received your letter of the 13th inst. together with a systematic collation of the collected work of the Committee

of the German Association for Testing Materials employed in Technology, and beg to thank you most heartily for the same.

It was with great interest that I examined the instructive document which forms, I have convinced myself a most appropriate scheme of arrangement.

As regards the arrangement prescribed in France, it appears to us, that is to say both to Mr. Becard and myself, after having fully considered the subject, that the time has not yet arrived to undertake re-editing and remodelling the tables which were transmitted to us some 18 months ago, as it is possible that the various methods of testing employed at the time by the authority referred to, may be combined; an official committee has at present taken the matter in hand as regards the behaviour of metals, after having carried to a conclusion similar researches regarding the behaviour of cements.

On the other hand the experiments which are now in progress in regard to brittleness, give grounds for the belief that these new methods of research will shortly be incorporated in contract specifications, so that it may be more advisable to await the final result of the experiments before attempting to collate the contract specifications usually prescribed in France.«

In reply to annother question dated the 30th January of the present year, the following answer was received:

»I beg you to excuse me for not having answered your letter sooner. I was waiting, however, to be able to inform you as regards the decisions arrived at in France by the official committee which has been entrusted with the revision of the contract requirements in general use«.

»As this committee has not yet stated the conclusions at »which it has arrived, it is impossible for me to give you any »further information in regard to this question, and I beg accor- »dingly to be excused«.

It is much to be desired that in so important industrial a country the committee should come to some conclusions which they can publish in the coures of the year.

Germany:	The German Association for Testing Materials employed in Technology has collected by its committee all the most representative contract specifications employed both in Germany and abroad, and carefully collated them. The work will be published in eight volumes. The committee has repeatedly considered the question of prescribing proposals of the kind asked for. It is very busily occupied with working them out and hopes to conclude its labours in this matter in the course of the year.
Russia:	In this country we have, alas, as yet no progress to report.
Austria:	In these countries the collection of data up to the year 1898 are now obtainable. The leading authorities have been requested to furnish the contract conditions prescribed during the last five years. The completion of the collection is now to be pushed on as rapidly as possible and a proposal will then be worked out.
Hungary:	Mr. von Tetmajer proposed to include in the collection of the standard specifications those of the R. Hung. Ministry of Trade, the R. Hung. State Railways, the I. R. Kassa-Oderberg Railway and the I. R. South Railway.
England:	The Committee of Engineers in London is, as reported by Mr. Unwin, not yet finished with the preparation of specifications.
Italy:	Mr. Guidi hopes to be able to make proposals within a short period.
Holland and Denmark:	Are agreed in general with the methods of procedure adopted in Germany and attempts are being made to formulate proposals in co-operation with the chairmen of the various committees in these states.

From Belgium, Luxemburg, Roumania, Sweden, Switzerland and Spain no communications have been received.

Nürnberg, April 1904.

Since my last report in April 1904 which required no alteration up to March 1st 1905, progress in the work of the committee may be reported.

Within the ambit of the programme of the committee to first collect representative contract specifications in various countries, and then to propose suggestions based upon them of such a kind as to be suitable for the basis of a uniform international proposal, separate suggestions have been worked out in North-America, England and Germany. From France and Sweden separate proposals or contract conditions are being shortly expected. Austria, Hungary and Italy are still employed in collecting the most important specifications in general use.

In particular the following is the general position of the work of the committees at present:

North-America: The Committee of the American Society for Testing Materials proposed in the course of the year 1904 and in September 1905 the following conditions for contracts for pig or cast iron and finished castings.

1. Standard specifications for foundry pig-iron,
2. „ „ „ cast iron-pipe and special castings,
3. Standard specifications for locomotive-cylinders,
4. „ „ „ cast-iron car-wheels,
5. „ „ „ malleable castings,
6. „ „ „ gray iron-castings.

These specifications of the American Society for Testing Materials, have been handed over to the Acting President of the International Association. The work of this committee would then in the main be finished.

France: The official committee which is engaged upon the determination of standard methods of testing has not as yet finished its labours; it has however, carried them so far that the publication

of standard specifications for railway-material may be expected shortly. The reporter of the French committee, Mr. Baclé, writes to me as follows under the 13th November 1905:

»In reply to the question which you have been good »enough to put to me, I have the honour of informing you »that the official committee entrusted with unifying the »contract-specifications for acceptance of materials for railways, »has not yet concluded its labours, so that it is not possible »to state what conclusions it will come to.

»The labours are nevertheless considerably advanced so »that it may be hoped that the publication of the unified »specifications will not be long delayed, and I shall not fail to »inform you when the publication takes place. I remain, etc.«

Germany: The following proposals have been made by the sub-committees of the German Association for Testing Materials employed in Technology:

1. Specifications for delivery or girders.

2. Standard specifications for delivery of component parts of iron bridges and skeletons in buildings drawn up by the Union of Architects and Engineers Societies, the Society of German Engineers, and the Society of Ironmasters.

3. Specifications of the Imperial Navy for testing and taking delivery of plates, section iron, rods, bolts and nuts, deck-beams, boilers, steel-plates and section steel for torpedo boats, steel-rods and bolts, open hearth-steel for boiler-plate, specifications of the Society of German Ironmasters regarding the allowable variations in plates or sheets less than 3 mm in thickness, as also

Specifications of the German Lloyd for testing puddled iron and steel.

4. Conditions for taking delivery for locomotive-tubes, tubes with copper packing, connecting tubes for ironwork, water-tubes for marine boilers, fire-tubes and holding-down-tubes for marine boilers.

5. Conditions for delivery of wire (extract from the specifications for delivery of iron and steel propounded by the Association of German Ironmasters.

Not yet completed is the work in regard to the following specifications:

6. For rolling stock, locomotives and carriages.
7. Boilers and high class sheet.
8. For cast-iron-tubes.

The above proposals 1 to 5 are subject to approval of next meeting of the German Association for Testing Materials employed in Technology. The council for the German Association has, however declared that it is willing that these proposals shall be laid before the International Association as an outline of what is required, on the understanding that the subject is to be completed. These „outlines of the German Association for Testing Materials employed in Technology to be decided upon at the next general meeting" together with the accompanying documents, have been handed over to the Acting President of the International Association.

Russia: In this country no progress is to be reported.

Austria: As regards the collection of the Austrian specifications, some of the most important of these have not been received and the chairman of the Austrian council or committee is engaged in the work of getting them in as quickly as possible.

Hungary: The work in this country has been interrupted by the decease of the Committee member M. von Tetmajer.

England: The English Engineer Committee in London (the Engineering Standards Committee) has already published the following normal contract specifications.

No. 2. Specification and sections for tramway-rails and fish-plates.

No. 8. British standard specification for tubular tramway-poles.

No. 9. British standard specification and sections for bull headed railway-rails.

No. 11. British standard specification and sections for flat bottomed railway-rails.

No. 13. British standard specification for structural steel for shipbuilding.

No. 14. British standard specification for structural steel for marine-boilers.

No. 15. British standard specification for telegraph material.

Further proposals were worked out by the Engineering Committee. The above seven specifications and the general report regarding the work of the Engineering Committee (Report on progress of work Jan. 1901—July 1905) have been sent to the Acting President of the International Association.

Italy: Mr. Guidi informs me that he has not yet received the contract specifications from the Italian State railway.

Holland and Denmark: These countries as already explained joined in the German procedure, the arrangement is that after the German proposals (see under Germany) have been placed before the committees in these countries, Denmark and Holland will adhere to them.

Sweden: The chairman of the Swedish committee, Mr. Deliner, has given us to understand that he will very shortly send contract specifications. As soon as I have received them I will place them in the hands of the International Association.

Luxemburg: The work of the Luxemburg committee has not proceeded very far, as Mr. Dufrux found himself compelled owing to residence in France, to retire from the committee.

From Belgium, Roumania, Switzerland and Spain no information has been received.

Nürnberg, May 1906.

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 2.

TO ESTABLISH METHODS OF INSPECTION AND TESTING FOR DETERMINING THE UNIFORMITY OF INDIVIDUAL SHIPMENTS OF IRON AND STEEL.

PRESENTED BY THE CHAIRMAN OF COMMITTEE 2, K. K. REGIERUNGSRAT
WILHELM AST,
BAUDIREKTOR DER K. K. PRIV. KAISER-FERDINANDS-NORDBAHN, VIENNA.
(TRANSLATED BY R. N. LUCAS BYFLEET.)

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To establish methods of inspection and testing for determining the uniformity of individual shipments of iron and steel.

Report presented by the Chairman of Committee 2
k. k. Regierungsrat **Wilhelm Ast,** Baudirektor der k. k. priv. Kaiser Ferdinands-Nordbahn, Vienna.

(Translated by R. N. Lucas Byfleet.)

The scope of the work of the Committee embraces the following three problems.

I. To determine methods of examining the homogeneity of iron and steel with reference to the ultimate utilisation of these methods in contract specifications.

II. To determine methods of examination by which the presence of those qualities which are required in steel and ingot-metal for special purposes can be easily and surely ascertained.

III. To decide on the value of the proposals of General Korobkoff in regard to a rational method of determining the tenacity of metals.

The problem No. 6 which was placed before the Committee at one time, referring to „The examination of the most suitable methods of polishing and etching wrought iron for the purpose of macroscopically examining the texture" was abandoned owing to the decision come to by the council of the Union at its tenth sitting on the 1st March 1902, to appoint Prof. E. Heyn of Charlottenburg to specially report on this subject

The members of the Committee were informed by the circular letter of the Chairman of November 1902, and also by those of April and November 1903 of the regulation referred to above. They were at the same time invited to further the objects of the Committee by studying the various subjects in the manner suggested in the programme agreed to at the congress of the Union at Budapest; and to furnish reports on their studies by 15th December 1903 at latest.

The invitation of the Chairman was accepted by the following members of the Committee: Engineer Le Blant, Paris, Inspector of the Eastern Railway of France, Chief Engineer Sauvage, Paris, Hofrat Professor Rejtö, Budapest, and Professor Hannover, Copenhagen; and they have sent in various exhaustive treatises. We take this opportunity to offer them our sincerest thanks.

The reports drawn up by Messrs. Le Blant, Sauvage and Hannover will be published as separate treatises in accordance with the decision of the Council at Vienna on the 9th Feb. 1904. Only excerpts therefore have been included in the Committee's report.

The following results refer to all the three problems with which the Committee has been engaged, and the individual reports are reproduced separately.

A. The results of the researches of the Chairman Regierungsrat W. Ast.

The material examined by the writer consists in the main of

a) 33 charges of basic open hearth mild steel N 1—33 of table I from the iron works A, from which small ingots were prepared and rolled down into flat bar of 30×15 mm section, and

b) 20 charges of basic mild steel 34—53 in Table I, ten being from the iron works B. (34—42 and 52) and ten from the iron works C. (43—51 and 53), the material being cast into large moulds and rolled down to plates 15 mm in thickness.

The tests to which this material was subjected consisted principally of

1. Macroscopic examination.
2. Static tensile tests, with prismatic rods.
3. Tests with notched rods, namely

a) Dynamic tensile tests.
b) Static bending tests.
c) Dynamic bending tests.

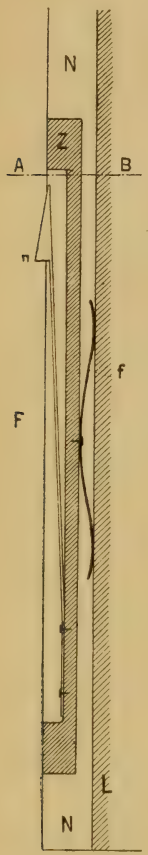
4. Bending test according to Korobkoff.
5. Ball pressure-tests.

The results of these tests are contained in table I, and are described in detail below.

1. Macroscopic tests in which hydrochloric acid is employed have been adopted as a form of preliminary test to ascertain the degree of impurity of the material. The results are given in column 2 of Table I. In charges 52 and 53 the material has been subdivided according to whether it consists of edge or central zone material; the test-rods being cut out of the plates so as to provide samples of the one or other material. The values given in the tables render the effect of liquation on the results of the tension-tests plainly visible. Further researches regarding the macroscopic structure of ingot-metal were not undertaken, as this subject was dealt with exhaustively by Committee II of the Association at the Budapest Congress; moreover a special treatise by the Reporter on „The results which have already been obtained by international research in the macroscopic examination of iron", which dealt with the subject in a very exhaustive manner, has been placed before the Congress.

2. Testing the material by static strain or tension was employed to determine its hardness and tenacity in a general way, and for the purpose of bringing the results of these tests into relationship with the results obtained under headings 3 to 5. These tests form the proper subject of the present report. The total elongation was ascertained on a basis of a measure of length (cm) of $\sqrt{80\,f}$, in which formula f corresponds to the section of the test-piece expressed in square cm. In addition the total elongation of the material in the portion of the test-piece held in the jaws of the testing machine was taken for a length of 5 mm,

so as to enable comparisons to be made with the bending test of Korobkoff, and was expressed in terms of the maximum elongation of the side of the test-piece under tension.



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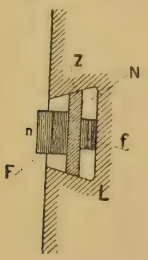


Fig. 1.

3. The tests with notched bars have reference, as already explained, to the static bending tests and the dynamic tension- and bending tests. For the purpose of carrying out the first of these a Mohr and Federhaff press was employed, while for the latter a percussion-apparatus constructed by the Reporter himself, and described in the report of the Committee to the Budapest Congress of 1901, was employed. In this apparatus the *vis viva* which the „monkey“ still possesses after the test-piece has been broken was registered by a spring apparatus. This percussion apparatus has undergone certain improvements in construction by means of which greater accuracy in the results obtained is assured. These improvements consist in means for registering the *vis viva* of the „monkey“ which has not been used up and for diminishing the friction of the rods in the spring apparatus.

The *vis viva* which is still possessed by the „monkey“ after the fracture of the test-piece is no longer, as was formerly done, measured by the extent to which the springs are compressed, but on the contrary is determined by the height to which the monkey is thrown up after compressing the spring. For this purpose in one of the two runners L of the percussion-apparatus, fig. 1, a vertical groove is arranged in which, owing to the tension of the spring F, the pointer Z is kept in its position while at the same time being easily displaced by friction. The spring F is attached to the indicator which, with the projection N, projects beyond the bearing surface provided for the runner and by means of light pressure is forced into the groove. The arrangement is carried out in such a way that the spring F of the descending monkey

presses in the groove, and in the moment at which it comes into contact with the apparatus shoots out the projection over the upper edge of the monkey. The rebounding „monkey" strikes against the projection *N* of the spring, carries this with it, and, together with the indicator-arm *Z*, permits it to remain in its highest position. The relationship between the height of the rebound and the corresponding *vis viva* is determined empirically.

The second important improvement of the percussion-apparatus consists in replacing surface friction in the rods of the apparatus by ball bearing friction.

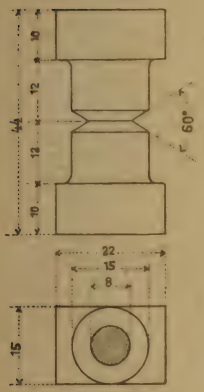


Fig. 2.

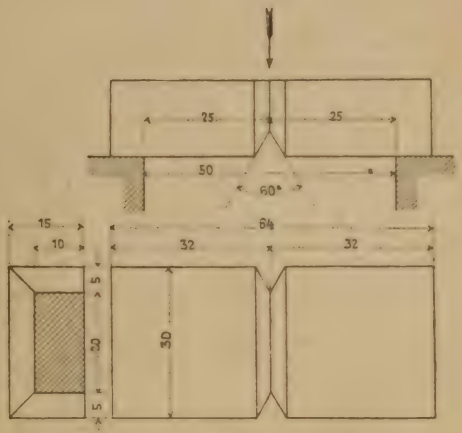


Fig. 3.

The percussion mechanism operates in its present form with great reliability and furnishes results of a degree of accuracy to fulfil the purpose for which the apparatus is designed.

In the dynamic tension-test the form of rod shown in fig. 2 was employed. The notch was in the first case cut with the knife and then finished off with the file, being left $^1/_2$ mm in width at its deepest part. The weight of the „monkey" was 5 kg, and the drop varied from 1 to 1·6 m according to the hardness of the materials. The rods were only subjected to the drop-percussion-test, and this did not fracture all. Some experienced only an unimportant amount of contraction, others on the contrary, displayed a very considerable amount of contraction. In table I are given

the amounts of energy absorbed instead of the work or energy required to produce fracture. This latter is indicated by an „*“. The values in the corresponding columns are in all cases expressed in cm^2 of cross section.

Fig. 3 shews the form of rod used in the tests. The notches were made in rods of the exact minimum size, viz 20×10 mm, according to the three following methods;

a) Milled with a milling-cutter, the deepest part of the notch being rounded off to a radius of $1^1/_2$ mm.

b) Filed to a width of $^1/_2$ mm at the deepest part of the notch.

c) Filed and cut in sharp at the deepest part of the notch.

All the rods were mounted on supports 50 mm apart, the notched portion of each rod being exactly between them. The force was applied in the plane of the smallest cross section, and at right angles to the unnotched side. The amount of force or work required for producing fracture was reckoned out per square centimeter of the diminished cross section and entered in table I. In the case of the dynamic bending test, in which a number of repeated blows were required to effect fracture of the test-piece, the effective work done has been included in the table, at full value. This method of calculating the expenditure of energy needed for fracture is, as a matter of fact, not quite correct, since at each blow a portion of the energy is consumed in elastic deformation of the test-piece; but it suffices in the present instance for the degree of comparison required.

The results of the experiments are shewn graphically and are collated both with one another and with the results of other methods of testing, for the purpose of ascertaining how far they obey any general law. In the sequel the results of those experiments will be communicated which have led to noteworthy results.

a) The diagram, figs. 4 and 5, illustrates the relationship subsisting between the strengths of the materials as ascertained by static tensile strength-test (No. 1—33) of Table I and expressed as abscissae, and the energy coefficient obtained in the dynamic tension-test or the bending test (till fracture occurs) expressed as ordinates. The latter are in the case of soft materials of higher value than in the case of hard materials. This diminution of resistance with increasing hardness of the material appears to be

smaller in the case of the tension-test than in the case of the bending test. The curve AB is the characteristic of rods with rounded notches; the curve CD is the characteristic of rods with sharp notches. The two different methods of preparing the sharp or pointed cuts have had but little influence on the result of the experiments, though in the case of rods that have been cut merely with the file the amount of energy absorbed is somewhat greater.

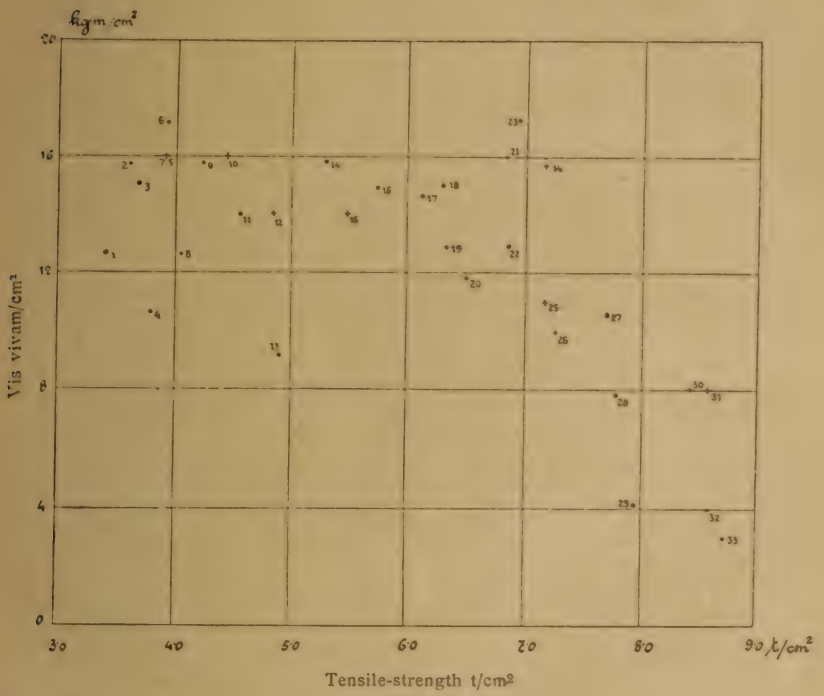


Fig. 4.

b) In agreement with the equivalent results obtained by Le Blant, it may be further seen from the diagram that the sharp cut has not so much effect in discriminating the hard materials which break easily, and which in this case are the harder, as the round cut has. For softer kinds of material the sharp cut is to be recommended for the purpose of limiting as much as possible the error which may occur in calculating out the amount of energy consumed when repeated blows have to be delivered in order to effect the fracture of the rod.

c) The relationships subsisting between the energy coefficients of the dynamic tensile test and the bending test with sharp, file-notched rods, can be seen from fig. 6, which has reference to the

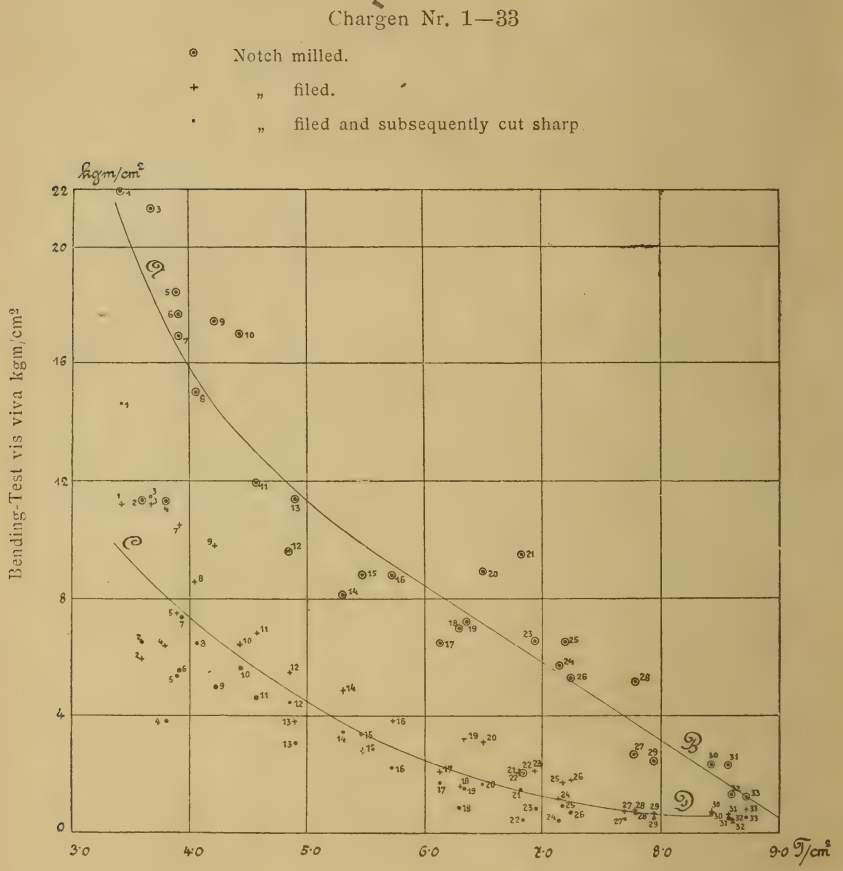


Fig. 5.

series of materials Nos. 1—33 of Table I. According to this illustration the more fragile materials are more effectively discriminated by the tensile test than by the bending test.

d) In fig. 7 the work coefficients of the dynamic bending test are given as abscissae, the angles to which the material is bent being given as the ordinates. The straight middle line AB shews that these two results stand to one another in a simple

relationship. This relationship applies to all the forms and methods of preparation of the notches in the rods. This is a very remarkable result, as, if generally true, it will enable the angle of flexure (up to the breaking point) to be used as a means of judging the quality of the material, which will of course produce a considerable simplification of the apparatus employed and the method of conducting the tests.

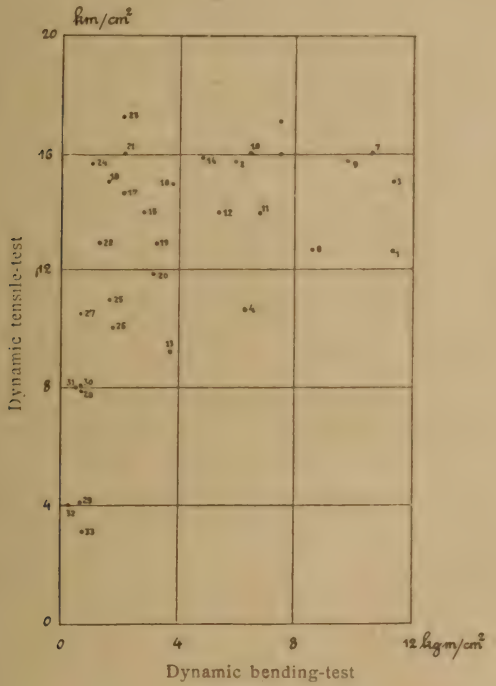


Fig. 6.

e) The value of the percussion bending test for determining the quality of the material is illustrated in a remarkable manner by fig. 9, in which the coefficients obtained for the samples 34—42 and 43—51 in Table I are brought into alternative relationship to one another. Of all the methods of testing, the percussion-bending test with sharply notched rods has brought out the greatest differences in the materials concerned. None of the other diagrams constructed by the writer shew a greater or more

remarkable regularity in mutual relationships of the various methods of testing materials. Further relationships may be found to subsist between the contraction-coefficients obtained by static tension-tests of prismatic rods and separate dynamic tests with notched rods.

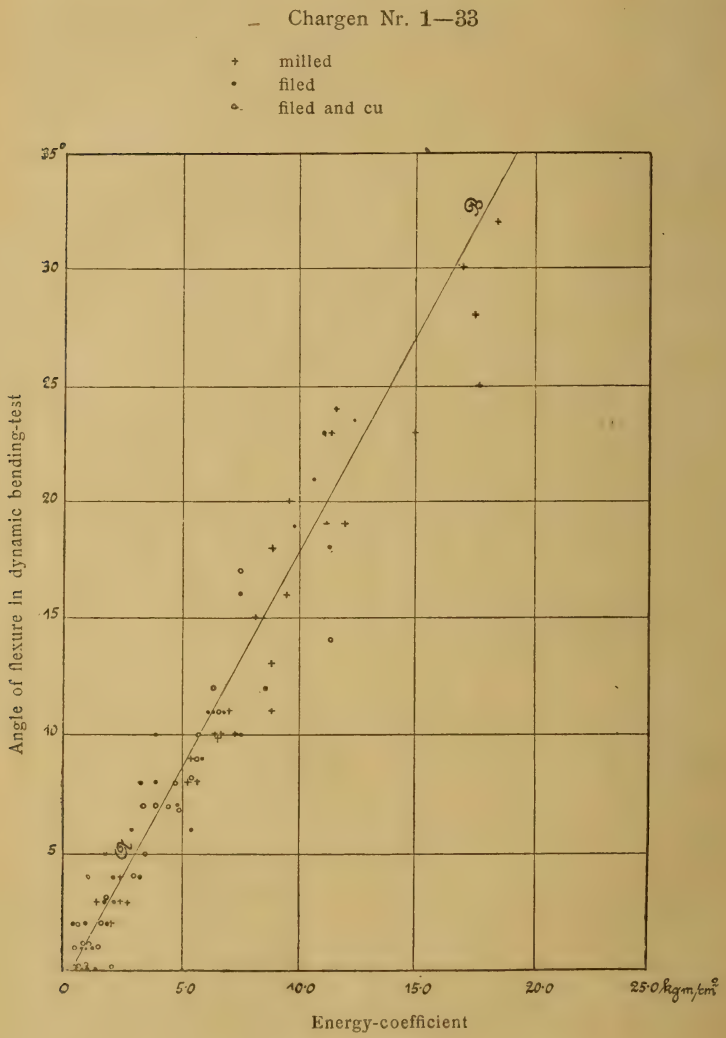


Fig. 7.

4. Bending tests according to the proposals of General Korobkoff.

All the test-pieces had the form shewn in fig. 8, that is to say the length was $1^1/_2$ times the thickness, as proposed by Korob-

koff, and the centre diminished to one half the cross section and marked every 5 mm on the side to be bent convex. In carrying out the experiment one end of the rod was fixed in a vice, and the other bent by a lever of suitable length. The bending of the ends of the test-pieces quite close together was also carried out in the vice. The elongation could be ascertained with great accuracy by means of a double pointed callipers with a magnification of 1—10. In addition the angle of the bend and the curvature-coefficient were also taken. The latter was calculated according to the formula $\frac{50 \times s}{r}$, in which s is the thickness of the rods and r the radius of curvature corresponding to the middle line mn.

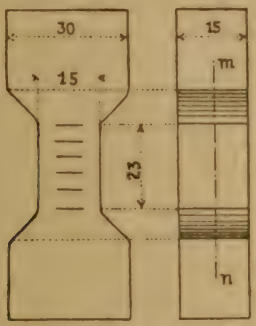


Fig. 8.

For purposes of comparison bending tests with prismatic rods were carried out at the Zürich Conference in 1895 by the permanent Committee No. IV in accordance with the suggestions of Sub-committee 13. The results of these tests, as also the above mentioned curvature-coefficients, are not included in table I. In what follows only those results will be described which were obtained with the material of charges Nos. 34—42 and 43—51 (table I). As these charges were from various works and obtained by different processes they enable a better comparison of the methods of testing to be obtained.

The experimental results obtained are shewn graphically in fig. 9, in which both the results of static tensile tests and particular tests with notched bars were brought into mutual comparison. The abscissae correspond in all cases to the tensile strength, the ordinates in

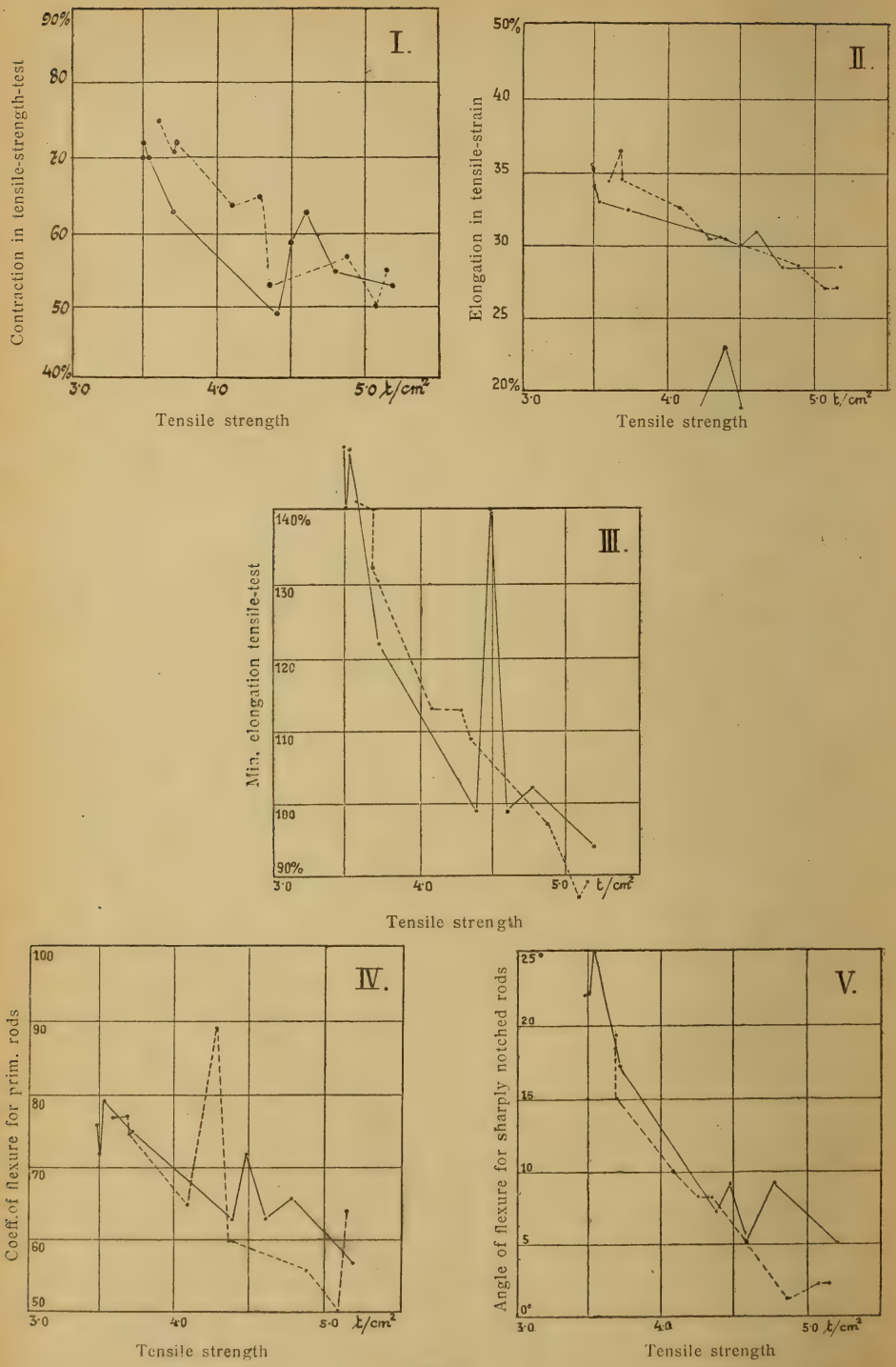

I.
Contraction in tensile-strength-test
90%
80
70
60
50
40%
3·0
4·0
5·0 t/cm²
Tensile strength
II.
Elongation in tensile-strain
50%
40
35
30
25
20%
3·0
4·0
5·0 t/cm²
Tensile strength
III.
Min. elongation tensile-test
140%
130
120
110
100
90%
3·0
4·0
5·0 t/cm²
Tensile strength
IV.
Coeff. of flexure for prim. rods
100
90
80
70
60
50
3·0
4·0
5·0 t/cm²
Tensile strength
V.
Angle of flexure for sharply notched rods
25°
20
15
10
5
0°
3·0
4·0
5·0 t/cm²
Tensile strength

Fig. 9.

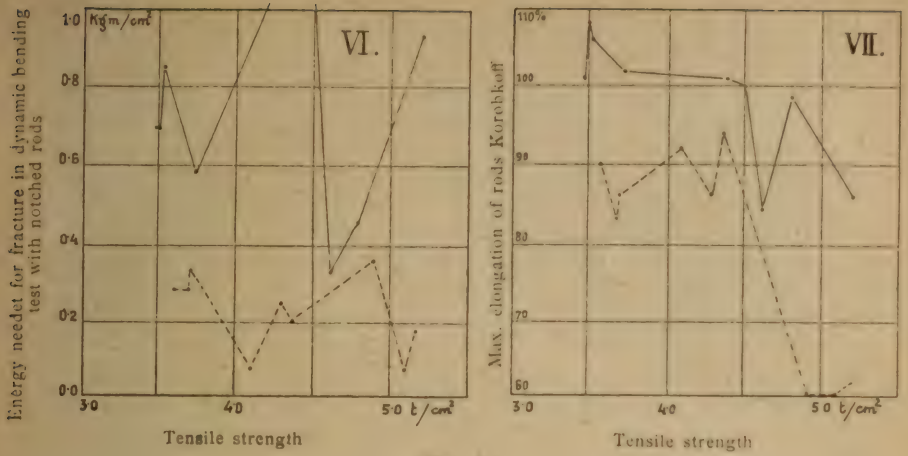
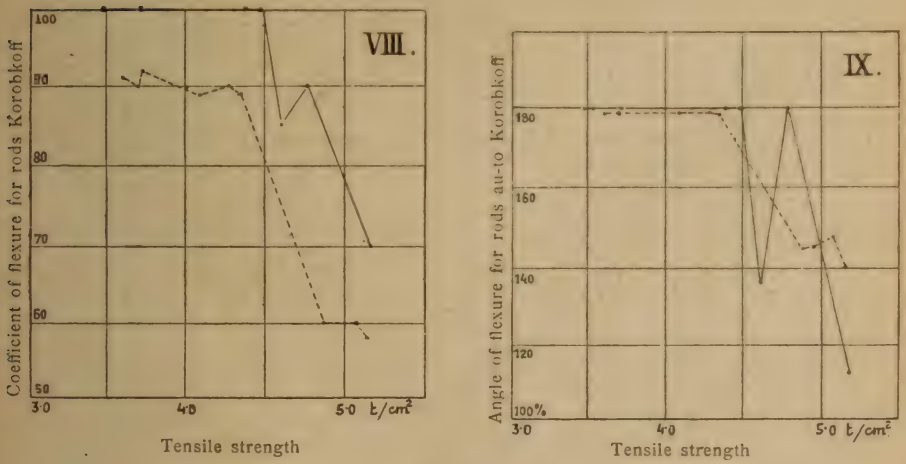


Fig. 9.

I. to the contraction obtained in the static tensile test,

II. to the total elongation in the static tension-test per 150 mm of length,

III. to the minimum elongations per 5 mm of the portion which underwent permanent contraction,



——————— Nr. 34—42
– – – – – – – – Nr. 43—51 } of Table I.

Fig. 9.

IV. to the curvature-coefficients obtained with prismatic rods in the static tension-test,

V. to the angles of bending obtained in the static bending test with sharply notched rods,

VI. to the coefficient of energy obtained in the dynamic bending test with sharply notched rods,

VII. to the maximum elongation obtained in the bending test according to Korobkoff per 5 mm,

VIII. to the curvature-coefficient obtained in the Korobkoff bending test,

IX. to the angle of flexure obtained in the Korobkoff bending test.

The full lines connect the results obtained with the series of materials Nos. 34—42, and the dotted lines the series 43—51.

From the illustrations I—V and IX no varieties of quality in the two sorts of materials employed can be perceived. It is otherwise with illustrations VI, VII and VIII.

All three allow the considerably inferior quality of the groups of materials 43—51 to be observed. This differentiation is not so apparent in the case of Korobkoff's bending test as in the case of the percussion bending test. It must, however, be observed that the Korobkoff bending test would have distinguished the softer materials better if the diminution in cross section of the samples were confined to a shorter length. The ductility of the material would in this case have been better able to manifest itself.

While therefore the static tensile test and the static bending test with prismatic rods has shewn no difference in the qualities of the classes of materials, the bending test proposed by General Korobkoff has effected this, and in the same sense as the percussion bending test with notched bars. The opinion, which is widely held, that the measurement of the elongation is difficult and also inaccurate cannot be shared by the writer, since he has obtained sufficiently accurate results with the reducing callipers he has employed. It must, however, be observed that the marks cut on the test-pieces in many cases give rise to premature injury and breakage, wherefore it would be advisable instead of employing the maximum elongations to employ curvature coefficients for comparison, and this has led to equally satisfactory discrimination of the materials. The essential principle of this method of testing involves the preparation of an adequate dimi-

nution in the cross section of the test-piece, this diminution not extending too far along its length. By this means the bending is confined to a small part of the test-piece, and the material is compelled to develop its full and special degree of ductility.

The results of the experiments herewith communicated correspond with the experience obtained by the writer in the case of other materials which he has examined, and the conclusion appears to be justified that the advantages of General Korobkoff's process are of general applicability. The great simplicity of the test is a further advantage, as no special apparatus is required for it.

5. The ball pressure-test, Brinell's system.

The means provided by this method of determining the hardness and tensile strength of iron at small cost and with little trouble have induced the writer to go into the subject more closely. For the method permits of the tests being multiplied in a simple way and so enables greater certainty in determining the hardness of the materials to be attained, both as regards single samples and large consigenments. The introductory experiments had for their object above all to determine what alterations the number V, which represents the ratio of hardness to tensile strength, is subject to when balls of various sizes, or loads of different magnitude, or materials of various degrees of hardness are tested. Brinell has pointed out the variations which occur when experiments are carried out under different conditions. No figures were given, however, and it was necessary to extend the test to other materials than those with which Brinell had experimented.

For the writer's experiments four samples of ingot-metal of different degrees of hardness were selected Care was taken to select only those that were free from liquation products, so as to obtain results influenced thereby as little as possible. The tensile strength of these four materials was 36.3, 51.5, 70.3 and 92.0 kg per sq. mm. The load on the test-pieces was applied by a Mohr & Federhaff press, the scale on which enabled exact readings to 10 kg and approximations down to 2 kg to be made. The determination of the diameter of the surface of pressure was effected by means of a microscope with micrometer eye-piece and tenfold magnification. In the case of the latter determination the 10^{ths} of milli-

meters can be read and hundredths easily estimated. For all the various kinds of materials, balls of 10, 30 and 50 mm in diameter were employed.

The results obtained are shewn graphically in figs. 10 to 13, in which the abscissae represent the diameter of the surface of pressure and the ordinates give the ratio-value V. All the results obtained with a given size of ball and a given material are connected by a continuous line marked with the diameter of the corresponding ball. It at once strikes the reader that the lines do not run regularly, and that large deflections occur such as one would not expect with an apparently equable material and adequate care in conducting the experiments. For this phenomenon no other explanation is forthcoming except difference in the material, and it has recently been proved that the results of the tensile tests only give the mean of the actual strengths of the materials.

The diagrams, figs. 10—13 show in what way, and to what extent, the ratio V is affected when balls of various sizes and different loads are employed; while the diagrams figs. 14—16 show the variations in the results of the experiments which are obtained with equal sized balls and equal loads when materials of different degrees of hardness are subjected to experiment. The design was at one time entertained of preparing a table to illustrate the relationships prevailing between the hardness of the material, the

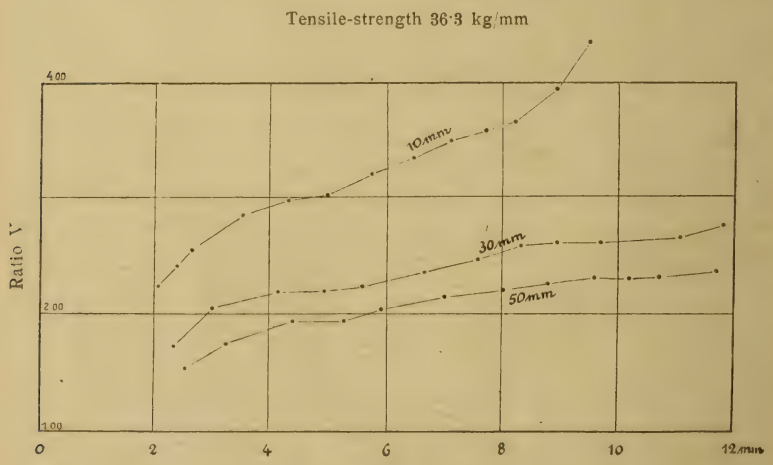


Fig. 10.

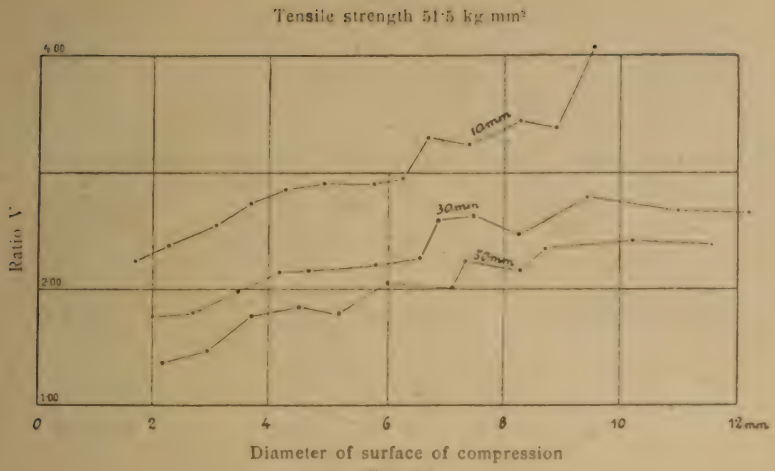


Fig. 11.

diameter of the ball employed, and the different loads applied. This idea has, however, been abandoned in view of the great variations in the results obtained. To arrange the results effectively in a table would have necessitated the writer finding a mean value for each test, and for this there was neither a sufficiency of time nor material. The diagrams furnished, will, however, suffice for a general survey of the question.

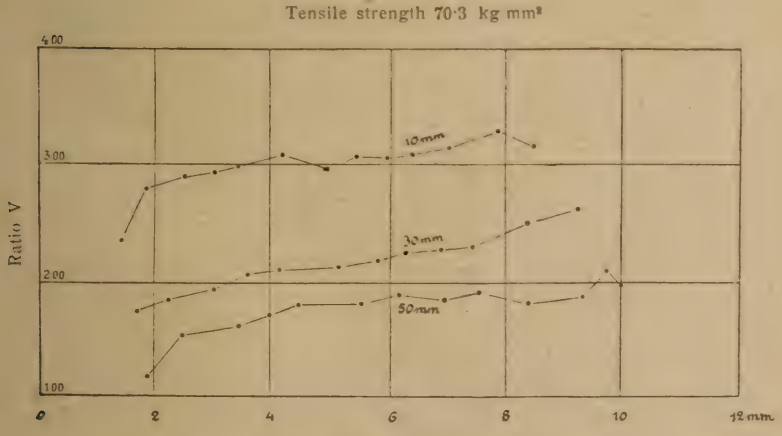
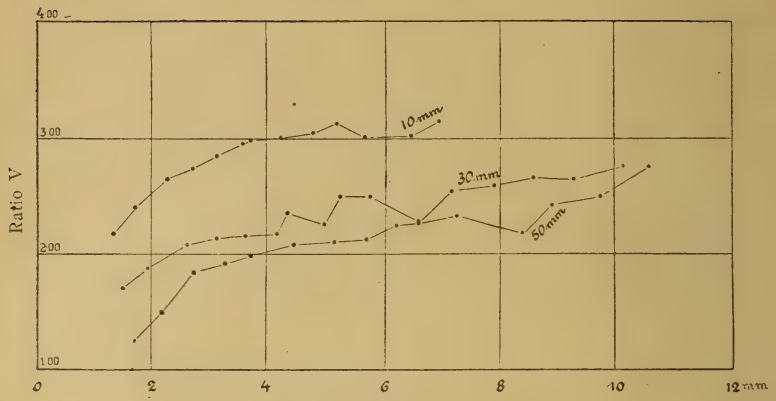


Fig. 12.

Tensile strength 92 0 kg/mm²

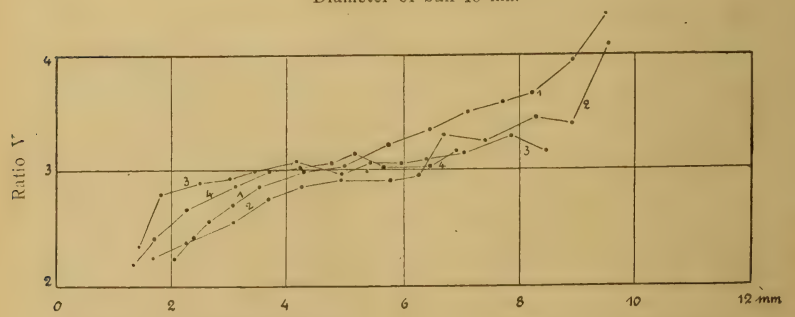


Diameter of surface of compression

Fig. 13.

As regards the size of the ball to be employed in any particular case, the results given proved that small balls even with very moderate loads give sharp edged pressure surfaces, whereas when large balls are used, very heavy loads are required to produce like results. For ordinary cases a diameter of ball of 30 mm may be regarded as the maximum limit. Smaller balls and smaller loads involve a higher degree of care in carrying out the experiments, if a satisfactory result is desired, whereas with larger balls and greater loads the same degree of care is not required. Under the latter conditions, too, the result is less

Diameter of ball 10 mm



Diameter of compression surface

Fig. 14.

1 . . . . 36·3 kg/mm² Tensile strength
2 . . . . 51·5 „ „ „
3 . . . . 70·3 „ „ „
4 . . . . 92·0 „ „ „

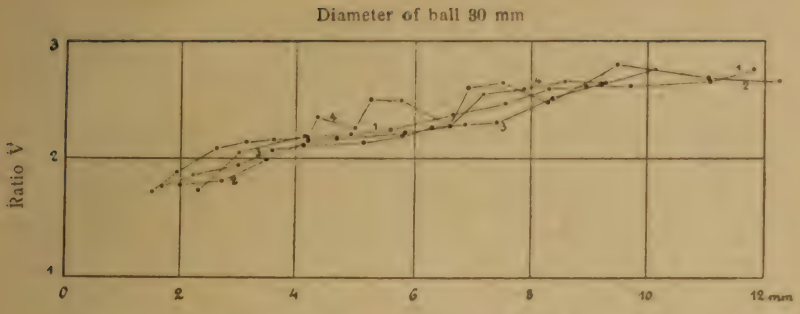


Fig. 15.

1 . . . . 36·3 kg/mm² Tensile strength
2 . . . . 51·5 „ „ „
3 . . . . 70·3 „ „ „
4 . . . . 92·0 „ „ „

affected by local irregularities in the composition of the material. When one is carrying out an experiment of this kind special attention must be bestowed on the conditions which prevail. In many cases a ball diameter 10 mm with a load of 2000 kg (about) will be suitable.

It was under the latter conditions that the hardness of the materials included in table I for the 33 charges of open hearth steel (or ingot-metal) were determined — two tests being made with each charge. The diameter of the pressure surface was 4·3—4·9 mm. In figure 17 the tensile strengths of the materials of the 33 charges are given as abscissae, the coefficients of hardness being represented by the ordinates. Each experiment is represented by a point. The straight line $A\ B$, which fits in satisfactorily between

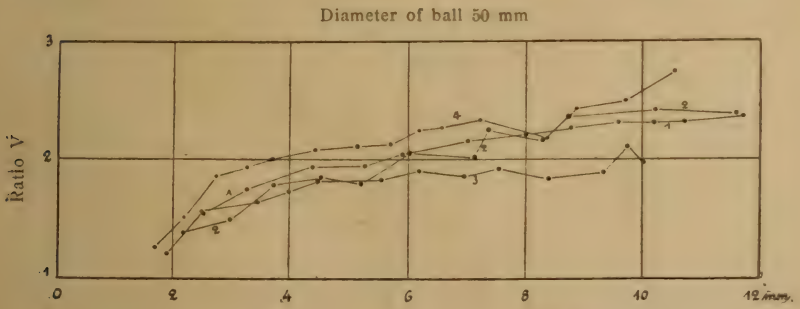
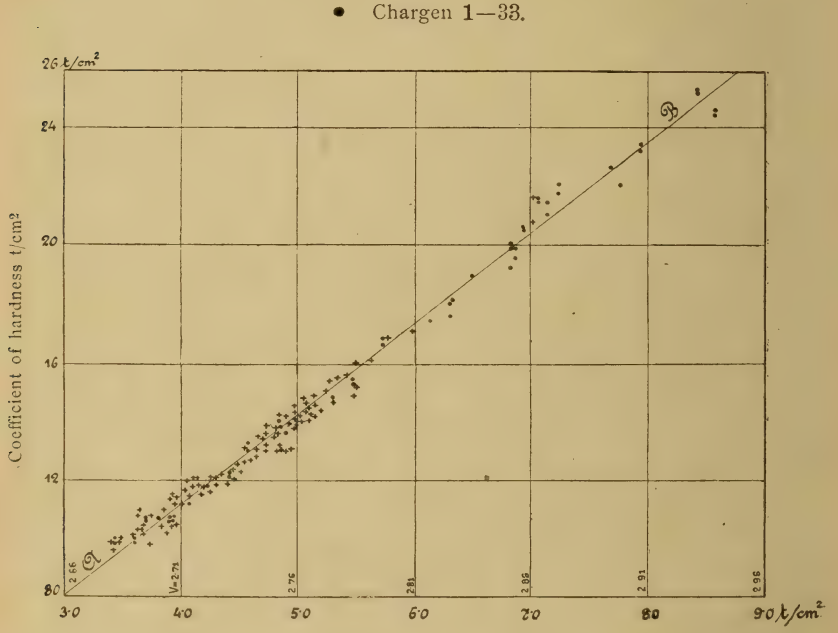


Fig. 16.

1 . . . . 36·3 kg/mm Tensile strength
2 . . . . 51·5 „ „ „
3 . . . . 70·3 „ „ „
4 . . . . 92 0 „ „ „

the results, gives the ratio-coefficient V entered on the corresponding ordinates. The ratio-coefficients increase in value with increasing hardness of material. The differences in hardness actually obtained never vary from the central line by more than a maximum of 10 kg per sq. mm. This is even true of the results, indicated by a cross, which were obtained with other materials. The tensile strengths obtained from the coefficients of hardness, would therefore



Tensile strength t/cm².

Fig. 17.

in no case vary more than about 3·5 kg per sq. mm from the tensile strengths obtained by the ordinary tension-test — a limit of error which is not too large for practical purposes. By repeating the experiments one can, however, determine the importance of the inequalities which exist, and also the mean tensile strength of the material, so that it differs from the true value to a much smaller extent.

The writer has employed the Brinell ball pressure-test with much advantage to determine the differences in the hardness

of the material due to the process of liquation in the ingot. Of the specimens examined two with characteristic central zone formation are referred to. These are two cylindrical shafts of 160 mm in diameter with edge zones of 20 and 40 mm. The surfaces were polished smooth with emery and then etched with hydrochloric acid to render the division between edge and central zones visible. The testing of the edge zone material took place at the edge of the section and in the neighbourhood of the line of solidification. The tests of the central zone material were made near the solidification line, then halfway between the line and the centre, and then near the centre. For each of these portions of the section the experiment was repeated 10 times.

In the figs. 18 and 19 the mean of each series of experiments is employed for the ordinates. The half sections of the shaft are introduced in plan. All the test-pieces tested have shewn the same relationships as are displayed in the two figures. The hardness is least at the edge of the section and increases almost uniformly towards the solidification line, at which it suddenly jumps up and remains nearly constant for the whole of that area. On the solidification line a harder ring (fig 19) of central zone material occurs.

Considering the relationships which subsist between the coefficient of hardness and tensile strength, the above relationships are also preserved by the latter quality.

Of all the substances combined with ingot-metal carbon is the element which possesses the greatest tendency to liquate (towards the centre of the ingot), and at the same time it is the element which tends most to confer hardness upon the metal. Figs. 18 and 19 therefore ought to be characteristic of the extent to which carbon has liquated, *i. e.* show the distribution of the carbon in the sections examined.

From the tests and experiments referred to it becomes clear that the ball pressure-test provides a simple and valuable method of testing for inequalities in the material. If, however, a thoroughly reliable judgment is to be formed as regards the hardness of the material, then it becomes much more important previously to determine the macroscopic structure and to repeat the experiments than in the case of the tensile strength test.

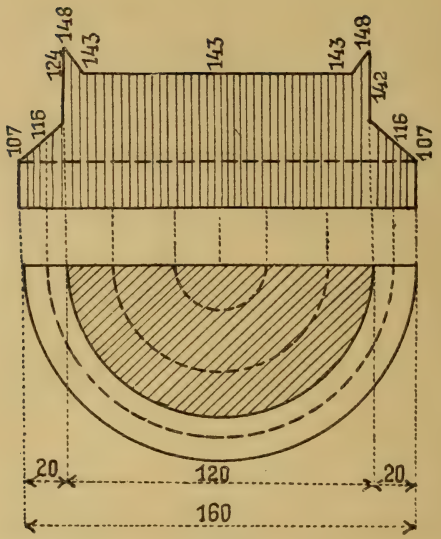


Fig. 18.

A glance at the tests of the material of the charges Nos. 1—33 shows that in all cases material of remarkably high quality was encountered. The cause of this phenomenon is mainly to be traced

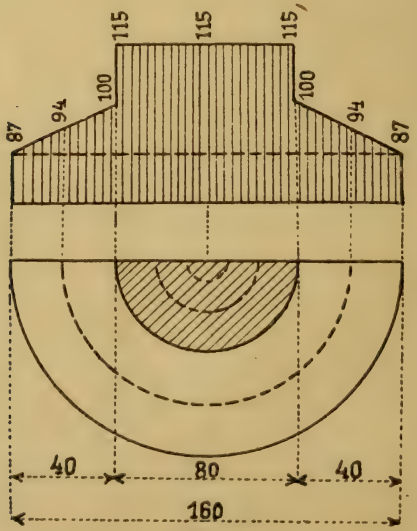


Fig. 19.

to the method of manufacture employed in preparing the steels from which the samples were taken, and to the manner in which it was carried out. It is also to some extent due to the small section to which the material was in each case rolled down, the temperature at which it passed through the finishing rolls being accordingly not too high. The results obtained with this material may be regarded in consequence as representing the maximum values attainable with ordinary mill-bars, and all the conclusions that may be drawn from them may be looked upon as constituting the characteristics of high class material.

B. Extract from the Report laid before the Congress by Engineer Le Blant, Inspector of the Eastern Railway of France.

The writer furnishes a report in regard to an extensive series of experiments carried out by himself on a variety of railway materials. These researches refer to new material and, in particular, material of a kind which has proved unsatisfactory in the workshops, or has given rise to accidents. The material was subjected to the customary tensile strength-tests with prismatic test-pieces, to the percussion bending test, and to the percussion-test with notched bars. These were of rectangular cross section and in two opposing sides were notched to a depth of one-sixth of the thickness of the bar in order to determine the influence of the method of preparation and form of the notches. Rods with round notches were tested in accordance with Charpy's proposals, i. e. rods were first bored out and then slit up. In the bending test the rods were either held at one end or mounted on two supports and struck.

The percussion apparatus employed by Le Blant was described by Engineer Barba at the Budapest Congress of the Association. It has been rendered more complete by the addition of an anvil which enables tensile strength-tests to be made without danger of causing bending or torsional strains.

The above named tests were mainly directed to settling the following questions:

1. Do the various methods of experiment furnish results comparable with one another?

2. What differences are displayed as regards the homogeneity of the material when bending tests and when tensile strength-tests are carried out?

3. Can contract specifications be framed from the results of the tests with notched test-pieces which have been carried out with material which has broken while in use?

Engineer Le Blant has graphically represented the results of his percussion-tests, and in this way has arrived at very interesting results. The curve in fig. 20 shows the influence which the kind

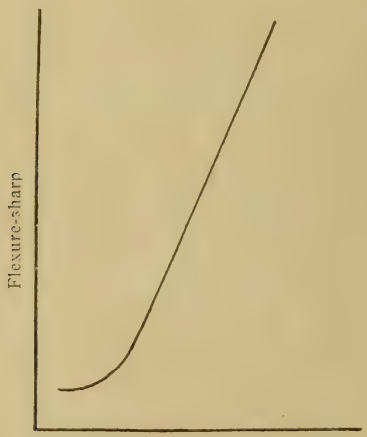


Fig. 20.

of notching has on the results of the bending test. Every point on the curve corresponds to a special material; its coordinates correspond to the values for the round (abscissae) or sharp (ordinates) notched rods. From this figure it may be seen that the sharp notch discriminates less between the more fragile materials than the less fragile, while in the case of the round notches the opposite is the case.

The curve fig. 21 which was similarly obtained illustrates the influence which the kind of test exercises on the result of the experiment when the rods have the same kind of notches, in

this case sharp ones. The coordinates here correspond to the energy coefficients which were obtained by the bending test (abscissae) or by the tensile strength-test (ordinates). From these illustrations it is clear that the bending test is best for discriminating between the more fragile materials; the tensile strength-test on the other hand is best for discriminating between the less fragile materials.

These tests enable us to decide which method of carrying out the percussion-test is the best in any particular case. Very fragile material should be subjected to the bending test with

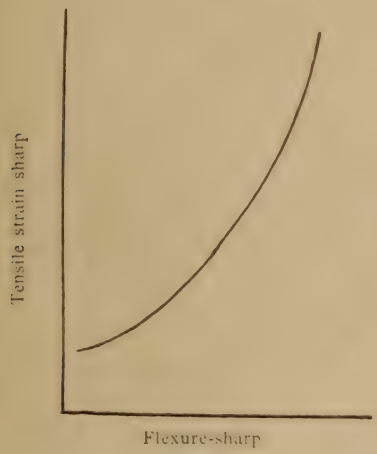


Fig. 21.

round notched rods, fragile materials to the test with sharp notched rods, and less fragile materials to tensile strain with sharply notched rods.

Testing the materials in regard to their variations in homogeneity has shewn that this quality is better brought into prominence by the percussion-bending test than by the percussion tensile strength-test.

The tests made by the East of France Railway have shown that difficulties arise when contract specifications are based on tests with notched rods or bars. The present position of testing for homogeneity is such that a material which has given test coefficients which stand to one another in the ratio 1 : 2, must

be regarded as thoroughly homogeneous, and it would be necessary to assign limiting values that would allow of a high degree of variation.

Engineer Le Blant concludes his report by expressing the view that the following rules should be observed for immediately determining standards:

1. The limit values must be placed so low as to lead to the selection only of better samples than the material which has broken while in use.

2. The experiments must be extended to various portions of the samples, and it must be understood that a single result that comes out below the minimum determined on should lead to the rejection of the sample.

C. Extract of the Report laid before the Congress by Chief-Engineer Sauvage.

Regarding the international results obtained from tests made with notched rods, Sauvage comes to the following conclusions as the result of his experiments:

For carrying out the experiments with notched rods a number of simple and practical forms of apparatus possessing a sufficient accuracy are available. These enable results comparable with one another to be obtained and may be employed indiscriminately.

The numerous experiments which have been carried out show no particular superiority of one process over another, and there is accordingly no reason to recommend one in preference to another. It is true that rods with rounded notches give different results from these in which the notches are sharp, but very special accuracy in making the notches hardly appears worthwhile. A saw-cut executed without any very unusual amount of care seems to suffice in the majority of cases.

In fact instead of one method of experiment having displayed any sort of superiority to or tended to displace the others, the general tendency of the experiments has been to introduce new and increased means of observation; such for example as the ob-

servation of the angle of bend after the fracture of the test-piece, alterations in the cross section (e. g. permanent contraction) at the point of fracture &c. New experiments are also being attempted, such as directing a series of blows along the length of a notched test-piece.

The expression in figures of the minimum amount of energy required to produce fracture has not been adequately determined in a practical manner, and it is for this reason that it has not been adopted in contract specifications.

D. Communications by Prof. A. Rejtö of Budapest on the fragility of materials.

The utility and practical value of materials can be determined on the basis of those characteristic qualities which are determined by the laws of Transmission of Force and of the internal friction, i. e. hardness tenacity and malleability.

On the basis of these qualities the behaviour of materials under any conditions, and in consequence, therefore, their fragility can, even in the case of notched rods subjected to percussion tests, be determined à priori.

In order to produce fracture, it is necessary that the tensile strain to which the material is subjected should equal the cohesive force (σ_0). In the case of malleable materials the internal friction is less than the cohesion, and as the external tensile strain, when the parts are free to glide, can only attain the value of that tensile strain (σ_e) that is necessary for overcoming the internal friction πf_e,*, the internal friction of malleable or plastic materials must be increased by artificially introducing forces, which we will call impeding forces or impedances (g), for the purpose of increasing the value of the cohesion.

The condition of fracture is, if F_b is the contracted cross-section and F_e the full cross-section before the fracture, that the

* See „Baumaterialienkunde Nos. 17/18 of 1904". „On the Value of Internal Friction" by A. Rejtö.

algebraic sum of the forces necessary for overcoming internal friction ($F_b \sigma_e$) and the impedance (g) shall be equal to the force required for overcoming the cohesion ($F_b \sigma_o$) i. e.

$$F_b \sigma_o = F_b \sigma_e + g$$

therefore

$$F_b (\sigma_o - \sigma_e) = g \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad 1)$$

The impedance is most simply occasioned by means of a surplus of material, which is not subjected to the effect of the lines of force caused by the tensile strain, being placed outside them. This is the effect in fact of notching.

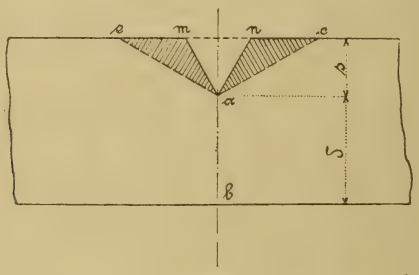


Fig. 22.

Let the notched rod be represented by fig 22. As soon as from any cause a tensile strain is produced at A the lines of force proceed in the direction $A\,c$ and $a\,e$. In these directions however, the molecules cannot move freely, as the parts of the material $a\,m\,e$ and $a\,n\,c$ operate as impedances. When the load (i. e. the tensile stress) is applied slowly, the impedance is proportional to the mass of material which so acts, and to the internal friction. The impeding mass of material is, in the case of bodies which obey the law of proportionality, proportional to the difference of section ε ($F_c - F_b$), to the depth of the notch (s), to the thickness ($a\,b = \sigma$), as also to the coefficient of internal friction (σ_e) and to the rate at which the load increases, that is to say, the internal friction is proportional to the maximum tensile strain. It is therefore

$$g = \varepsilon (F_e - F_b) \, . \, \sigma_c \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad 2)$$

In the case of percussive stresses, the molecules lying in the direction or line of action, have their rate of movement accelerated, and as this acceleration depends upon the terminal velocity (v) of the drop hammer, the resistance is not σ_e but

$$\sigma_e [1 + f(v - v_o)]$$

if (v_o) represents that velocity at which the value of σ was determined.

In general therefore

$$g = \varepsilon (F_e - F_b) \sigma_e [1 + f(v - v_o)] \quad . \quad . \quad . \quad . \quad . \quad 3)$$

Substituting this value for g in formula 1 we have

$$F_b (\sigma_o - \sigma_e) = \varepsilon (F_e - F_b) \sigma_e [1 + f(v - v_o)]$$

or

$$\frac{\sigma_o - \sigma_e}{\sigma_e} = \varepsilon \frac{F_e - F_b}{F_b} [1 + f(v - v_o)]$$

as however

$$\frac{\sigma_o}{\sigma_e} = B,$$

i. e. the degree of maleability is

$$B - 1 = \varepsilon \frac{F_e - F_b}{F_b} [1 + f(v - v_o)] \quad . \quad . \quad . \quad . \quad 4)$$

if $v = v_o$

$$B - 1 = \varepsilon \frac{F_e - F_b}{F_b} \quad . \quad . \quad . \quad . \quad . \quad . \quad 5)$$

The above formula is fully applicable in the case of tensile and shearing stresses, and gives the degree of contraction in tensile strain-tests and the fragility in the case of shearing strain-tests.

To decide the practical applicability of this deduction in the case of percussion-tests with notched rods, I requested the Chairman of Committee No 2, Councillor W. Ast, to send me samples of those materials which he had subjected to percussion tests with notched rods. Councillor Ast was good enough to accede to my request and also forwarded me the results of his experiments.

The tests-pieces he was good enough to send me were, however, of such a size that only two or three tensile tests with notched rods, as well as ordinary tension and compression-tests,

could be made. I should have been very glad to make in addition tests for shearing stress, but unfortunately I had not sufficient material for this.

The result of my tensile tests are appended in the following table II, in which at the same time I have included the tensile tests of Councillor W. Ast so as to facilitate comparison. The results agree absolutely except in two cases. Sample No 20 gave in Councillor Ast's, tests 86 kg, but in mine 96 kg per mm^2, while No 32 according to Ast gave 77·8 kg and in my experiments 73·4 kg. In these two cases I have taken Ast's results into account. Councillor Ast carried out his percussion-tests with notched rods in such a way that the mass of the test-piece and the notching to which it was subjected were identical. $\frac{F_e - F_b}{F_b}$ is accordingly constant; in addition the drop-hammer was always raised to the same height $H = 1{\cdot}5$ m. So that the velocity of each blow was constant $= 2{\cdot}42$ m, consequently the expression $f\ (v - v\,o)$ in formula 4 is also constant.

In formulae 4 and 5 the values on the left side are dependent on the qualities of the material, and the other side of the equations must consequently be variable in correspondence with those qualities.

Councillor Ast carried out his experiments in such a way that the masses of the test-pieces and their notches were always the same, so formulae 4 and 5 are accordingly not applicable in this case.

In the communications already referred to on „The Value of Internal Friction" and „The Determination of Characteristics by Shearing Stress", I have demonstrated that the fragility depends on the value $(B - 1)$ in the case of tensile strain and shearing stress-tests. In percussion-tests with notched bars it also probably depends on $B - 1$.

In order to determine this, it was necessary to ascertain the ratio of malleability to the energy of percussion which caused fraction in the tests by Councillor Ast.

As, however, a ratio can only be established in the case of quantities of the same dimension, it must first be decided whether the expression $B - 1$ is a linear or square value.

In those shearing tests which I employed for determining the characteristics, the width of the test-piece is unaltered and is accordingly $\frac{F_e - F_b}{F_b} = \frac{\delta_e - \delta_b}{\delta_b}$ from which it follows that in this case $B - 1$ is a value of linear dimension.

In tensile tests the whole cross-section is altered. The extent of this alteration can be determined by means of the constancy of volume as follows:

When the increase in length which corresponds to the contraction occurring with equable extension is calculated, and if this is termed h_b, the constancy of volume can be determined as follows:

$$F_e h_e = F_b h_b$$

from which

$$F_b = F_e \frac{h_e}{h_b}$$

substituting this in $\frac{F_e - F_b}{F_b}$

we get

$$B - 1 = \varepsilon \frac{F_e - F_e \frac{h_e}{h_b}}{F_e \frac{h_e}{h_b}}$$

therefore

$$B - 1 = \varepsilon \frac{h_b - h_e}{h_e}$$

consequently $B - 1$ in the case of tensile tests is a ratio value of linear dimension.

On this account we must assume that $B - 1$ maintains the same character even with notched bars, that is to say, it is a ratio value of linear dimension; as, however, the percussion A is a cubic value, it can only be brought into the relation with a cubic value of $B - 1$, therefore

$$(B - 1)^3 = \eta A \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad 6)$$

as, however,

$$A = GH$$

in which H is the drop and G the weight, which was constant in all cases,

$$(B-1)^3 = (\eta \,.\, G)\, H$$

from which

$$B = \sqrt[3]{\eta G}\,.\,\sqrt[3]{H} + 1$$

and if

$$\sqrt[3]{\eta G} = \eta_0$$

then

$$B = \eta_0\,.\,\sqrt[3]{H} + 1. \quad \ldots \quad 7)$$

The coefficients η and η_0 depend on the velocity of the blow, on the form and depth of the notch and on the ratio of external and internal labours. They must, therefore, be determined for any kind of effects.

The values for η_0 obtained by formula 7, are represented as functions of the necessary heights in fig. 23, from which it may be seen that the value of η_0 diminishes with the number of blows as follows:

No. of blows	1	2	3	4
Mean value of No.	1·07	0·875	0·776	0·760

The value of η_0 could not be determined in the case of 4 blows, as only one value was known. It is probably about 0·760.

The values calculated by means of formula 7 for malleability, are inserted in table II, from which it may be seen that as regards percussion tests, they agree closely with the results obtained by means of tensile strain tests.

All the results obtained by Councillor Ast being thus in agreement with the plasticity, I proceeded to deal with the results of the experiments carried out by Mr. Vanderheym.

After the Budapest Congress I sent to Mr. Vanderheym two specimens of ingot-metal Nos. 157 and 156, with the request that he would be good enough to carry out the necessary experiments and acquaint me with the results. Mr. Vanderheym kindly did as requested and supplied me with the following results:

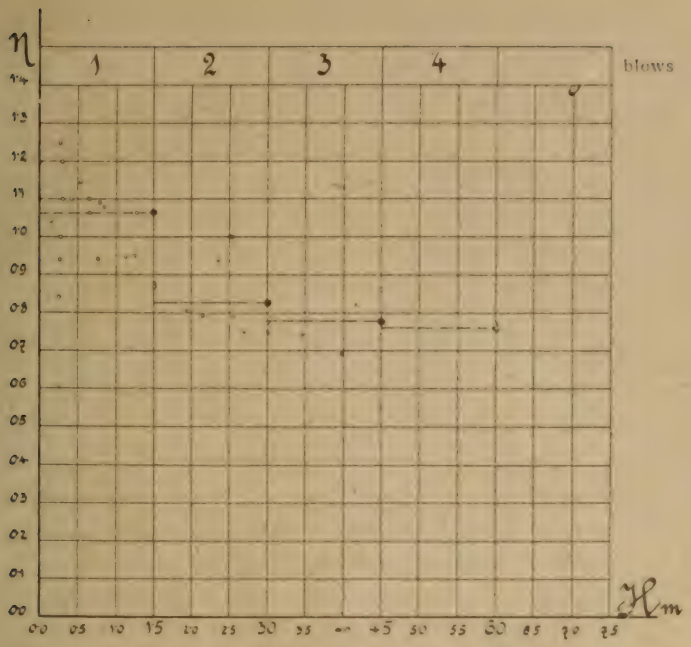


Fig. 23.

For No 157 : $H = 1{\cdot}5 - 1{\cdot}3$ m

„ „ 156 : $H = 0{\cdot}5 - 0{\cdot}45$ m

from which

„ „ 157 : $\sqrt[3]{FH} = 1{\cdot}31 - 1{\cdot}19$ m

and „ „ 156 : $\sqrt[3]{FH} = 0{\cdot}793 - 0{\cdot}766$ m

since from test

„ „ 157 : $B - 1 = 1{\cdot}08$ m

and „ „ 156 : $B - 1 = 0{\cdot}66 - 0{\cdot}7$ m

are obtained, so it is obvious that the values obtained by Vanderheym's method agree with the malleability.

From the results given it follows that:

1. The fragility of notched rods is no inherent quality of the material, but depends on the malleability.

2. The malleability should be determined in all cases in which the form of construction involves unequal distribution of the masses, or blows or shocks are likely to be experienced, since the resistance to stresses and strains of this kind increases at the 3rd power of $B - 1$.

3. Percussion-tests with notched bars are particularly valuable for determining the malleability, and should be especially recommended in contracts for large quantities of materials.

4. These experiments provide a general view of the homogeneity of the materials. The want of homogeneity in the materials occasions in the 1st instance maximum and minimum values, i. e. limits of cohesion by which limits of the malleability are called for; as, however, the energy expended to produce fracture in the case of percussion-tests with notched rods is proportional to the 3rd power of the malleability, their limiting values also increase in proportion to the 3rd power of the malleability $(B - 1)$, which enables the degree of homogeneity of the materials to be clearly seen.

Conclusions.

Problem I. The inequalities of material due to liquation are considered explained. This is particularly the case in regard to the practical aspects of the question, while those aspects of it which are of a purely scientific nature have been less completely solved. Further study, particularly from the latter standpoint, is accordingly to be recommended.

Knowledge of the nature of material homogeneity, as revealed by the experiments with notched rods, have recently been greatly enriched by the studies of eminent experimenters. On the other-hand there has been a great difference of opinion regarding the value of the experiments in general and of the percussion-tests in particular, more especially as regards the conditions under which the experiment should be carried out, and whether the experiment brings to light a new quality of the material or not. In spite of the wide difference of opinion it appears to have been established that the percussion bending test, with sharply notched rods, provides a means of distinguishing the qualities of materials which has not been so far approached by any other methods,

and this must be regarded as a practical result of the highest order. To clear up the subject and remove the prevailing differences of opinion, extensive studies under the most widely varying conditions will be necessary.

Problem II. Numerous experiments recently carried out have led to the perfection and development of newer methods, and have essentially enriched our knowledge of the qualities of materials. Thus, for instance, the simple determination of hardness on the Brinell, and tenacity on the Korobkoff methods, and the considerable improvements in the methods of determining homogeneity (particularly the etching test and test with notched rods) have greatly contributed to the solution of problem II. A complete solution will, however, only be possible after the studies for question 1 have been concluded.

Problem III. The experiments of the writer give ground for the assumption that the tenacity of the material is rendered more apparent by the bending test proposed by General Korobkoff, than by the usual tensile and bending tests with prismatic rods. As measure of the tenacity the maximum dislocation of the grain of the material and, in the second place, the coefficient of flexure are recommended. The verification of the results by means of experiments with materials from other sources must be regarded as desirable.

Table I.

No.	Etching test	Tensile test: static with prismatic rods: Permanent elongation kg/cm²	Tensile strength kg/cm²	Contraction %	Elongation per original length: 150 mm %	Elongation per original length: 5 mm in contracted part of rod %	Tensile test: dynamic with notched rods: Energy needed to produce fracture kg/cm²	Bending test with notched rods: static: milled: Breaking strain kg/cm²	milled: Break. angle degree	filed: Breaking strain kg/cm²	filed: Break. angle degree	filed and cut: Breaking strain kg/cm²	filed and cut: Break. angle degree	dynamic: milled: No. of blows	milled: Energy needed to prod. fract. kg/cm²	milled: Break. angle degree	filed: No. of blows	filed: Energy needed to prod. fract. kg/cm²	filed: Break. angle degree	filed and cut: No. of blows	filed and cut: Energy needed to prod. fract. kg/cm²	filed and cut: Break. angle degree	Korobkoff's bending test: Bending angle degree	Max. elongation per 5 mm original length %	Brinell's ball pressure test 10 mm ball 43—49 mm pressure diametre of surface of pressure: Coefficient of hardness kg/cm²	Ratio of hardness and tensile strength
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27
1	pure, Lunker	2220	3410	71	347	168	1270	1845	—	1575	—	1685	—	6	2200	—	3	1125	18	4	1458	—	180	90	9800	288
2	pure	2350	3600	63	333	142	1580	1725	27	1520	18	1560	14	4	1163	24	2	593	9	2	645	11	180	88	9900	275
3	pure, Lunker	2540	3690	66	353	116	1510	1860	—	1750	—	1710	—	6	2130	—	4	1125	23	3	1138	14	180	96	10600	287
4	pure	2400	3800	70	360	114	1070	2045	37	1835	18	1820	18	3	1125	19	2	630	11	1	375	7	180	94	10700	282
5	pure	2610	3900	67	326	138	1600*	1985	36	1765	14	1680	12	5	1840	32	2	750	10	2	530	8	180	90	10500	277
6	pure	2380	3910	67	320	142	172	2135	23	1830	10	1810	12	5	1770	25	2	750	16	2	553	9	180	102	10800	276
7	pure	2440	3910	70	346	146	1600*	2025	30	1880	15	1975	—	5	1693	39	3	1055	21	2	750	17	180	94	10900	279
8	pure, Lunker	2600	4060	59	333	122	1270	1895	22	1575	—	1725	14	4	1500	23	3	858	12	2	638	12	180	92	11200	275
9	pure	2770	4220	62	300	128	1580	2165	28	1910	10	1850	—	5	1750	28	3	980	19	2	488	7	180	104	11800	280
10	pure	2560	4430	61	300	122	1600*	2075	—	1780	—	1840	18	5	1700	30	2	638	11	2	558	10	180	100	12200	275
11	pure	3150	4570	58	293	100	1400	2135	15	1935	7	1825	–	4	1195	19	2	678	11	2	463	8	180	106	13000	284
12	pure	3020	4850	57	273	96	1400*	2035	18	1770	10	1815	9	3	963	20	2	540	6	2	435	7	180	106	14000	288
13	pure	2970	4900	54	273	98	920	2140	12	1750	—	1780	9	4	1135	23	1	375	10	1	295	4	180	104	13600	277
14	pure, Lunker	3160	5300	54	253	104	1590	2010	18	1665	8	1790	15	3	813	15	2	480	7	1	340	7	180	104	14800	280
15	pure	3400	5480	53	260	84	1400*	2030	15	1795	4	1725	7	3	880	13	1	275	6	1	330	5	180	102	15400	281
16	pure	3490	5730	54	253	96	1500	22~0	9	1825	5	1825	10	3	883	18	1	375	8	1	213	0	180	110	16800	293
17	pure	3200	6120	51	240	88	1470	2225	12	1760	6	1760	4	2	635	10	1	207	4	1	165	3	180	94	17600	287
18	pure	3670	6300	58	260	110	1510	2360	12	1915	4	1600	2	2	700	11	1	158	3	1	080	0	180	102	18000	285

19	pure	3830	6320	60	174	108	1290	2525	10	2005	6	2120	7	2	725	10	1	315	4	1	105	1	180	102	18100	286
20	pure	3930	6500	60	213	92	1190	2570	12	2100	6	2075	3	3	893	11	1	313	8	1	155	2	180	106	18900	290
21	pure	3780	6820	57	213	92	1600	2655	12	2090	6	2040	5	3	950	16	1	213	3	1	140	1	180	112	20000	293
22	pure	4180	6860	50	200	82	1290	2480	11	2045	4	1650	1	1	203	2	1	130	0	1	043	0	180	108	19900	290
23	pure	4120	6950	54	200	100	1730	2400	10	2045	5	2050	2	2	655	10	1	208	4	1	080	0	180	108	20500	295
24	pure	4150	7150	55	180	96	1570*	2585	8	2080	4	2045	3	2	565	8	1	108	1	1	043	0	180	102	21400	299
25	pure	4390	7170	54	213	94	1100*	2620	10	1975	4	2050	2	2	653	10	1	165	5	1	093	4	180	104	21500	300
26	pure	4200	7250	52	187	68	1000*	2600	10	2015	4	1975	2	2	525	9	1	183	2	1	070	1	180	106	22000	304
27	pure, Lunker	4520	7700	49	133	82	1050	2340	7	1950	3	1760	3	1	270	3	1	078	0	1	043	1	180	114	22600	294
28	pure	3980	7780	40	160	58	790	2270	10	1970	7	1955	0	2	515	8	1	075	2	1	083	0	180	112	22000	283
29	pure	4700	7950	42	180	64	410	2575	4	1815	0	1695	0	1	245	—	1	065	1	1	045	0	180	118	23400	295
30	pure	4910	8430	44	160	66	800*	2995	3	1815	0	—	—	1	283	3	1	068	1	1	068	0	180	122	25300	300
31	pure	4650	8580	26	140	28	800*	2480	5	1980	4	1860	0	1	285	4	1	063	1	1	045	2	93	42	24600	287
32	pure	5200	8600	33	107	54	400	2440	1	1975	1	1075	0	1	128	3	1	033	2	1	038	0	148	50	29500	343
33	pure	5430	8720	28	120	34	310	2225	2	1650	0	1725	0	1	130	3	1	078	0	1	048	0	180	122	26300	300
34	pure	1940	3490	70	355	148	—	2015	80	1875	27	1845	22	1	083	2	1	043	1	1	068	0	180	101	10000	287
35	pure	2180	3520	70	330	148	—	1980	78	1930	23	1840	25	1	095	0	1	070	0	1	085	0	180	106	9800	278
36	pure	2030	3500	72	340	140	—	2065	80	1985	25	1715	22	1	095	0	1	075	0	1	068	0	180	108	9800	280
37	pure	2360	4490	59	300	104	—	2010	19	1590	10	1530	9	1	073	4	1	058	2	1	103	0	180	100	12400	276
38	pure	2670	5180	53	285	94	—	2085	18	1715	9	1730	5	2	363	15	1	075	2	1	093	0	113	86	14000	270
39	pure	2460	4390	49	305	99	—	2105	20	1665	5	1530	7	3	1058	15	1	088	1	1	120	3	180	101	12000	273
40	pure	2690	4780	55	285	102	—	2100	20	1730	7	1635	9	1	055	0	1	088	0	1	043	0	180	99	13500	282
41	pure	2420	4600	53	310	99	—	2095	22	1570	7	1440	5	1	045	0	1	040	0	1	083	0	137	84	12600	274
42	pure	2120	3710	63	325	122	—	2075	64	1825	20	1600	17	1	070	0	1	045	1	1	058	1	180	102	10400	280
43	impure	2320	3680	71	365	132	—	2320	75	2010	22	1805	19	1	058	0	1	038	0	1	028	0	180	83	10500	285
44	impure	2270	3590	75	345	141	—	2310	73	2070	28	1715	18	1	048	0	1	028	0	1	028	0	180	90	10200	284
45	impure	2390	3690	72	345	140	—	2365	63	1980	20	1740	15	1	025	0	1	025	0	1	033	0	180	86	10300	279
46	impure	2580	4090	64	325	113	—	2345	30	1875	16	1655	10	1	040	0	1	023	0	1	008	0	180	92	11700	285
47	impure	2710	4270	65	305	123	—	2475	38	1735	11	1435	8	1	030	0	1	018	0	1	025	0	180	96	12000	281
48	impure	2780	5150	55	270	89	—	2185	15	1400	4	1330	2	1	043	0	1	038	0	1	018	0	180	100	14000	272
49	impure	3130	4890	57	285	97	—	2160	14	1600	7	1280	1	1	030	0	1	058	0	1	038	0	142	60	13800	281
50	impure	3010	5090	50	270	87	—	2155	12	1600	7	1240	2	1	035	0	1	030	0	1	008	0	147	60	14100	277
51	impure	2580	4340	63	305	109	—	2375	25	1390	9	1535	8	1	020	0	1	035	0	1	020	0	180	94	12200	281
52	edge	2410	4070	65	330	122	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	11500	283
	kernel	2980	4970	53	262	108	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	14300	287
53	edge	2150	3430	72	355	148	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	9400	273
	kernel	2490	4090	55	318	132	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	11500	281

Table II.

No. of test-piece	Results of H. Regierungsrat W. Ast				Results of H. Hofrat Rejtö			Calculated degree of ductility	Difference between calculation and experimental blow %
	Tensile strength kg/mm²	Number of blows	Excess of drop of last blow m	Total effective drop m	Tensile of strength original	Tensile of strength altered	Degree of ductility		
					cross section kg/mm²				
1	34·1	3	0·00	4·50	33·75 33 80 — 33·78	41 0 41·12 — 41 06	2·425	2·29	— 5·5
2	36	2	0·63	2·37	34·65 36·8 — 35·72	42·22 44·4 — 43·31	2·24	2·1	— 7
3	36·9	4	0·00	6·00	37·0 36·2 — 36 6	45·8 43·5 — 44·65	2·41	2 39	— 0·8
4	38	2	0·48	2·52	36·5 37·0 — 36·75	44·0 44·8 — 44·4	2·375	2·125	— 12
5	39	2	0·00	3 00	41·0 39·55 — 40·27	51·0 47·6 — 49·3	2·126	2·19	+ 3·5
6	39·1	3	0·28	4·22	37·7 38·32 — 38·01	45·2 46·0 — 45·6	2·325	2·26	— 3
7	39·1	2	0·00	3·00	39·2 38·5 — 38·8	47·5 46·95 — 47·23	2·190	2·19	+ 0
8	40·6	3	1·07	3·43	38·0 39·3 — 38·65	45·75 47·0 — 46·37	2·12	2·17	+ 2·5
9	42·2	3	0·58	3·92	41·45 41·35 — 41·40	49 15 50·5 — 49·83	2·09	2 22	+ 6
10	44·3	2	0·45	2·55	43·2 43·5 — 43 35	52·0 52·72 — 52·36	2·07	2·125	+ 2

No. of test-piece	Results of H. Regierungsrat W. Ast				Results of H. Hofrat Rejtö			Calculated degree of ductility	Difference between calculation and experimental blow %
	Tensile strength kg/mm²	Number of blows	Excess of drop of last blow m	Total effective drop m	Tensile of strength original	Tensile of strength altered	Degree of ductility		
					cross section kg/mm²				
11	45·7	2	0·29	2·71	44·5 44·25 — 44·37	53·22 53·35 — 53·285	2·06	2.155	+ 5
12	48·5	2	0·84	2·16	48·85 — — 48·85	58·4 — — 58·4	2·02	2·07	+ 2·2
13	49·0	1	0·00	1·50	46·5 49·6 — 48·05	55·3 59·0 — 57·2	2·01	2·23	+ 11
14	53·0	2	1·08	1·92	50·55 51·73 — 51·14	59·5 60·8 — 60·15	2·00	2·02	+ 1
15	54·8	1	0·40	1·10	51·95 52·5 — 52·72	62·1 63·1 — 62·6	1·985	2·1	+ 5·5
16	57·3	1	0·00	1·50	57·10 57·40 — 57·25	65·5 67·2 — 66·35	2·01	2·23	+ 11
18	63·0	1	0·87	0·63	— 63·0 — 63·0	— 71·6 — 71·6	1·94	1·91	— 1·5
19	63·2	1	0·24	1·26	61·3 62·2 — 61·75	70·2 70·25 — 70·23	2·162	2·16	— 0·1
20	65·0	1	0·25	1·25	64·5 64·5 — 64·5	74·5 74·7 — 74·6	2·027	2·15	+ 6
21	68·2	1	0·65	0·85	67·0 68·6 — 67·8	76·3 77·1 — 76·7	2·02	2·01	— 0·5
22	68·6	1	0·98	0·52	66·78 68·5 — 67·64	75·47 76·50 — 75·98	1·91	1·855	— 3

No. of test-piece	Results of H. Regierungsrat W. Ast				Results of H. Hofrat Rejtö			Calculated degree of ductility	Difference between calculation and experimental blow %
	Tensile strength kg/mm²	Number of blows	Excess of drop of last blow m	Total effective drop m	Tensile of strength original	Tensile of strength altered	Degree of ductility		
					cross section kg/mm²				
23	69·5	1	0·77	0·73	68·9 68·1 — 68·5	77·4 78·0 — 77·7	1·981	1·96	− 1
24	71·5	1	1·07	0·43	71·0 70·6 — 70·8	80·5 78·3 — 79·4	1·82	1·8	— 1
25	71·7	1	0·84	0·66	72·3 71·5 — 71·9	80·75 80·5 — 80·63	1·932	1·93	— 0·1
26	72·5	1	0·77	0·73	72·1 72·0 — 72·05	80·75 81·5 — 81·12	1·847	1·96	+ 6
27	77·0	1	1·21	0·29	77·7 75·9 — 76·8	86·75 83·70 — 85·225	183 (1·725)	1·71	− 7
28	77·8	1	1·20	0·30	73·9 73·0 — 73·45	82·4 81·0 — 81·7 (87·5)	1·775 (1·67)	1·75	— 1·5
29	79·5	1	1·24	0·26	81·6 — — 81·6	88·8 — — 88·8	1·635	1·67	+ 2
30	84·3	1	1·23	0·27	85·5 86·0 — 85·75	92·7 93·4 — 93·05	1·597	1·68	+ 5
31	85·8	1	1·25	0·25	84·4 84·0 — 84·2	92·0 93·0 — 92·5	1·53	1·675	+ 9·5
32	86·0	1	1·37	0·13	95·2 97·6 — 95·9	102·5 106·5 — 104·5 (93·0)	1·355 (1·52)	1·53	+ 0·7
33	87·2	1	1·19	0·31	89·5 83·0 — 86·25	96·7 90·6 — 93·65	1·83	1·72	— 6

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 6.

REPORT ON METHODS OF ETCHING MALLEABLE IRON FOR THE VISUAL INVESTIGATION OF STRUCTURE; AND ON THE LESSONS TO BE LEARNT THEREFROM.

COMMUNICATION FROM THE ROYAL TESTING LABORATORY AT GROSS-LICHTERFELDE, W., NEAR BERLIN. BY

PROF. E. HEYN.

(TRANSLATED BY F. H. LEEDS, F. I. C., LONDON.)

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Report on methods of etching malleable iron for the visual investigation of structure; and on the lessons to be learnt therefrom.

Communication from the Royal Testing Laboratory at Gross-Lichterfelde, W., near Berlin.

By Prof. **E. Heyn.**

(Translated by F. H. Leeds, F. I. C., London.)

Having been requested to prepare an account of the lessons that may be learnt as to the structure of iron and steel by inspecting etched surfaces of those metals, the author now submits the following report, in which attention is principally bestowed upon the results obtainable from a mere examination with the unassisted eye. It should, however, be remarked that the microscope must be employed to gain a thorough understanding of the appearances presented; and that all other aids to research, such as analysis, mechanical tests upon small samples, etc., must be made use of, if the causes and effects of the characteristics brought to light by etching are to be properly understood. It is also advisable to employ such etching reagents as allow of a subsequent microscopic examination, for only in that way can the investigator avoid the danger of drawing erroneous conclusions. Hence the necessity for preparing photomicrographs cannot be altogether obviated if the results of a visual examination are to be trustworthy.

The author is unable to recommend the use of hydrochloric acid, which is so often adopted as the etching fluid. As will be shown later, it eats certain constituents of the metal away and so makes holes. Moreover, owing to hollow places in the original sample, or to depressions produced during the action of the acid, it undercuts the material and comes into contact with a large extent of surface, until deep cavities are made to appear where there were none, or where only trivial flaws existed at first. Again, samples etched with hydrochloric acid cannot be subsequently examined under the microscope. To avoid all these inconveniences, cuprammonium chloride has been adopted at the Testing Laboratory as the etching reagent, a solution being prepared by dissolving 1 gramme of the solid in 12 grammes of water. When special circumstances do not call for an alteration in the rule, transverse sections of the metal are prepared with a thickness of about 10 millimetres. They are ground with emery cloths of different degrees of fineness glued to revolving wood discs, and are finally polished on cloth-covered buffs with rouge and water. The preparation usually occupies about two hours, but when more extensive surfaces have to be examined (as in the case of tall I-girders or «Grey» beams[1] the time taken is correspondingly longer. When, e. g., the transverse section of a very tall I-girder has to be investigated, it should be cut in two, each half being ground and polished separately. With its prepared surface upwards, the sample is then quickly dropped (see fig. 1) into the etching

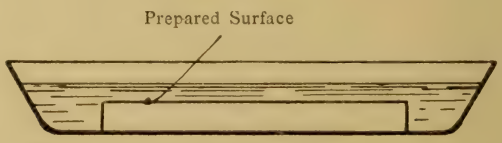

Fig. 1.

fluid in a large glass or papier-maché dish. The introduction of the sample into the liquid requires a certain amount of practice if lines and markings are to be avoided, the operation being performed in a manner resembling the immersion of a photographic

[1] The «Grey» girder is one possessing specially wide flanges, and is commonly referred to in England as having a «Differdingen section». — Translator.

plate in its developer. A formation of bubbles must also be prevented, or parts of the metal will be protected from the fluid, and small spots will be left in relief which reflect light. The etching takes about one minute. Owing to the reaction between the iron of the metal and the copper in the solution, spongy copper separates upon the ground surface, and is removed by rubbing it with a pad of cotton wool after the sample has been transferred to a vessel of water, through which a constant stream of fresh water is preferably kept flowing. If the concentration of the etching liquid is as specified (1 : 12) the copper is very easy to remove; but if it is too weak, the copper adheres firmly, rendering the etching operation unsuccessful. Too highly concentrated solutions act too quickly and powerfully and exhibit no advantage. When the washing is complete, the sample is rinsed in a little alcohol, and carefully dried by dabbing it with a soft handkerchief — it should not be rubbed lest the surface be injured. So prepared, the specimens keep fairly well without rusting in ordinary air, and may be preserved indefinitely in an atmosphere dried with calcium chloride. A beginner, who has not attained sufficient dexterity to avoid markings and irregular etching, should polish and etch his samples a second time, in order to see if the peculiarities of the surface are characteristic or due to imperfect preparation. If intrinsic, they will remain sensibly the same; if extrinsic, they will be different.

Cuprammonium chloride is not suitable for the etching of cast iron containing graphite, for the galvanic action of the latter causes the deposited copper to adhere firmly to the surface. It can, however, be used with perfect success in the treatment of white cast iron and ordinary carbon steels, especially when they are not hardened. The copper adheres firmly to the surfaces of special steels, such as contain much nickel or chromium; and therefore the nature of these steels becomes manifest during the etching operation. Special steels must accordingly be etched with other fluids.

The different structural components of iron-carbon alloys are attacked in a very similar fashion by cuprammonium chloride. Holes are not eaten away, nor appreciably increased in size by the etching. If, therefore, cavities or microscopic depressions are noticed on the etched surface, they must have been there originally,

and are not the result of the etching process. This is a notable advantage possessed by cuprammonium chloride over hydrochloric acid. Minute pores or cavities in iron are easily closed up by polishing, thus becoming invisible before the surface is etched. Some time after the metal has been etched with cuprammonium chloride, however, the cavities are rendered very distinct, for liquid (especially the alcohol used for rinsing the surface) exudes from them. Such exudation may occur repeatedly, and if the liquid is not wiped away, marks may be produced in the vicinity of the pores which an unpractised investigator may confuse with the dark colorations due to other causes that will be described hereinafter. In photograph 13, the dark rays leading to the edge of the picture are rows of fine pores; but they are surrounded by still darker margins, the result of exudations.

Etching by means of cuprammonium chloride is very useful in detecting spots of high carbon or phosphorus content; also in bringing into notice permanent deformations the metal may have suffered in the cold.

a) Distribution of the Carbon. — Spots of high carbon content in unhardened malleable iron appear dark after etching. This is due to the fact that pearlite is darkened by cuprammonium chloride, whereas ferrite remains uncoloured. Parts of the metal richer than others in carbon look darker, therefore, because they contain more pearlite. The effect may be seen in photograph 1, which is the representation of a piece of steel superficially decarburized by prolonged heating. The margin conists of an iron with an amount of carbon that is practically nil. The margin encloses a darker core containing the original high percentage of carbon. As a surface etched with cuprammonium chloride is suitable for examination under the microscope, it may advisedly be submitted to such investigation in order to obtain confirmatory evidence of the irregular distribution of the carbon. From the ratio of pearlite and ferrite, it is even possible to judge of the amount of carbon present in different parts of the prepared surface: that is to say, to carry out carbon estimations under the microscope. This is specially important when the effect of tempering or case-hardening has to be ascertained. In the latter instance the depth of the carburised layer and the approximate amount of carbon in it can be

judged. The sample should be etched before being chilled in water, for the chilling renders an estimation of the carbon content impossible. When it is desired to examine the carburised portion of a sample that has already been chilled, it must first be heated, preferably in a bath of molten metal, so that loss of carbon by oxidation may be prevented.

Microscopic investigation is a very delicate method of determining whether the superficial parts of an ingot contain an amount of carbon different from that in the interior of the mass. Micrograph 2 (× 365) represents the structure at the surface of a rolled girder; micrograph 3 (also × 365) the structure of the interior. The preponderance of dark pearlite masses in the micrograph of the interior portion shows at once the greater proportion of carbon which exists there in comparison with that at the edges. Both micrographs have been prepared from hand-drawings, in which certain markings due to other causes have been omitted.

b) Appearance of spots rich in Phosphorus.[1] — Photograph 4 is the representation in its natural size of a sample of iron prepared in the following manner: A fragment of phosphoritic metal (S 644, 6) containing 0.08 per cent of carbon, 0.27 per cent of manganese, and 0.28 per cent of phosphorus (corresponding in fact with a Thomas-Gilchrist metal before complete dephosphorisation) was artificially inserted in the centre of a piece of steel (S 660) containing only 0.01 per cent of phosphorus, 0.07 per cent of carbon, and 0.10 per cent of manganese: the whole being ground and etched for one minute with cuprammonium chloride. The phosphoritic centre assumed a dark colour with the suggestion of a bronzelike tint, whereas the purer margins remained pale. The perfectly black spots are cavities in the core. After a little practice it is possible to distinguish between the dark coloration of the core due to high phosphorus content and the dark colour [as mentioned under (a)] produced by much carbon; but in cases of doubt the microscope can be referred to. For the purpose of comparing the effects of different etching fluids, photographs 5 and 6 are submitted. The metal in the former was etched with 1 : 5

[1] See „Einiges aus der metallographischen Praxis“ (Notes on metallographic Practice), by E. Heyn. Stahl und Eisen, 1906, page 8.

nitric acid, and exhibits no difference in colour between the centre portion and the margins. In photograph 6 the metal was etched for 3·5 hours with a mixture of 3 parts of 1·19 hydrochloric acid and 7 parts of water. This liquid energetically attacked the phosphorised core, but left the less highly phosphoritic margins relatively untouched. Hence the core is roughened, fails to reflect light as strongly as the margins do, and consequently appears dark. The undercutting action of hydrochloric acid is clearly exemplified in photograph 6. The upper right corner of the central portion has completely come away, while the fissure leading to the centre from the lower left edge has been seriously widened. Thus etching by means of hydrochloric acid prevents faulty spots in the sample from being estimated correctly, and causes them to be exaggerated. The difference in the solubility of materials rich and poor in phosphorus was determined by some special tests. Two pieces of the above-described varieties of steel containing (S 660) 0.01 per cent and (S 644, 6) 0.28 per cent of phosphorus respectively were placed in a beaker together, with hydrochloric acid of the strength already mentioned, and allowed to remain for 3·5 hours at ordinary room temperatures, the liquid being frequently stirred up. The losses in weight were as follows: —

	low P (S 660)	high P (S 644, 6)
Before treatment	55.6024	24.2600
After treatment	55.5194	17.7800
Loss in weight (grammes) .	0.0830	6.4800

Allowance being made for the somewhat smaller area of the sample rich in phosphorus, it will be seen that the ratio of solubility is at least 1 : 80. Hence, when steel is etched with hydrochloric acid, the spots of high phosphorus content are eaten away; and, at the places where a segregation of the phosphoric constituent has occurred, a cavity is formed of much greater dimensions than correspond with the actual amount of such segregation. The higher speed of the etching process in the depressions of the sample increases its exposed area, so that the metal is attacked in the neighbourhood of the segregated portions more violently than elsewhere. The expression „porous steel“ was formerly much used to describe such etching phenomena, but to adopt it is to confuse

cause and effect. Hydrochloric acid is quite unsuitable for the detection of small cavities that are not already visible before etching. For this particular object cuprammonium chloride is indispensable, inasmuch as it does not make holes but only produces different colour effects.

The darkening action of cuprammonium chloride upon spots rich in phosphorus depends on the fact that crystals of ferrite containing iron phosphide in solid solution (i. e. mixed crystals of iron and iron phosphide) are coloured more deeply than spots which contain less or no phosphorus. This is presumably due to a formation of copper phosphide upon the prepared surface under the influence of the etching fluid Pure iron phosphide, on the contrary, is not attacked by cuprammonium chloride, remaining pale in colour like cementite, the iron carbide; and since ferrite is only able to retain in solid solution a little over 1 per cent of phosphorus in the form of phosphide, the darkening action of cuprammonium chloride ceases when that limit is reached. This is exemplified in micrograph 7 (× 123) which represents a portion of incompletely dephosphorised Thomas-Gilchrist metal (S 644, 2) containing 1·78 per cent of phosphorus, 0·17 per cent of carbon and 0·19 per cent of manganese The light-coloured veins are iron phosphide, surrounding dark mixed crystals of ferrite saturated with phosphide. As is frequently to be observed with such mixed crystals, their central portions contain less phosphorus, and the proportion of that substance increases in the vicinity of the iron phosphide veins, as is evident from the pale colour there. Thus cuprammonium chloride solution only darkens crystals of ferrite that contain less than a certain proportion of phosphorus, the critical amount being about 1 per cent.[1] In malleable iron, such a limit is not likely to be reached, even when much segregation has taken place — at least the author has not yet succeeded in observing it. The cuprammonium chloride reaction for phosphorus fails in the case of highly carburised alloys of iron where ferrite is absent and its place taken by cementite. Other tests, however, can be applied, but since they are purely microscopic, they will not be dealt with now.

c) Recognition of cold-worked spots — When cuprammonium chloride is used to etch a piece of iron that

[1] See «Iron and Phosphorus» by Stead, J. Iron and Steel Inst., 1900.

has undergone more severe permanent deformation in one place than in another, the spots which have suffered most appear the darker — more particularly if the stresses were of a tensional or shearing character. This may be seen in photographs 8 and 9 which represent longitudinal sections through two cylinders of steel originally 20 millimetres in diameter and 20 millimetres in height after being upset. The sample in photograph 8 has been decreased in height by 8·7 millimetres, and that in photograph 9 by 14 millimetres through the treatment. The effect is most noticeable in the diagonals, which therefore appear dark after etching. The cause of the deeper colouring may be recognised under the microscope. In the direction of the stress furrows of some microscopic depth and size have been eaten away during the etching operation, and their number locally diminishes the power of the prepared surface to reflect light. Hence the spots seem darker to the unassisted eye. A further indication of the deformation will be found in the shape of the iron granules, which are no longer equiaxial but elongated.[2] Photograph 10 represents the foot of an I-girder with a hole punched through it. The punch (cf. fig. 2)

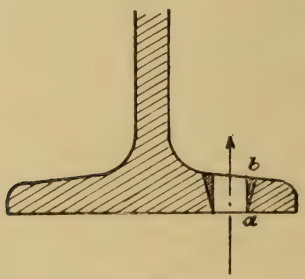

Fig. 2.

has caused a movement of the metal in the direction of the arrow. Owing to the deformation that has taken place during the punching, when the prepared surface is etched with cuprammonium chloride, it appears dark along the circumference of the hole, as at $a-b$.

[2] The author has already dealt exhaustively with this matter. See „Die Umwandlung des Kleingefüges bei Eisen und Kupfer durch Formveränderung im kalten Zustande und darauffolgendes Ausglühen“ (Alterations in Structure of Iron and Copper produced by Cold Deformation and subsequent Heating), by E. Heyn. Zeitschr. des Vereines deutscher Ingenieure, 1900, Nos. 14 and 16.

The deeper tint produced in this way cannot be mistaken for the darker colours due to high carbon or phosphorus content (as described above) if the spot is examined more carefully under the microscope. It then exhibits a structure resembling that shown by micrograph 11 (× 365), where dark etching furrows lie parallel to the elongated ferrite particles.

Having thus described the general effects produced by etching with cuprammonium chloride, a few examples of the application of the process to the examination of materials may be given.

The degree of homogeneity among the different chemical components of iron as exhibited by the section of a forged or rolled ingot depends primarily upon the manner in which the liquid metal has solidified; for the segregation that has occurred during that process cannot be remedied by any subsequent treatment. Through the courtesy of a manufacturer, the author has been provided with a set of sections from ingots and from girders rolled from them, whence it has been possible, by employing the etching process, to follow the successive changes that have taken place in the structural character of the metal. From this collection the following example may be quoted. The ingot was obtained as the result of a special run in a Thomas-Gilchrist converter.[1] Photograph 12 represents in its natural size one-quarter of a transverse section from the top of the ingot after etching with cuprammonium chloride, photograph 13 being similar, but taken from the bottom. An I-girder was rolled from the ingot, and photograph 14 shows an etched section from the top end, while photograph 15 represents the bottom, both pictures being of natural dimensions. The deep black markings in 12 and 13 are cavities. Two areas are clearly to be distinguished in photograph 12 (top end), a dark central portion and a paler margin, a still darker band separating them. The same appearance is noticeable in the corresponding girder photograph (No. 14): viz., a very dark band separating a darkish centre from a pale margin. At the bottom end of the ingot (photograph 13), the different areas are less distinct, and the pale margin is no longer separated from the slightly darker tinted centre by a deep

[1] This fact is mentioned explicitly in order that the discussion here followed for purely scientific purposes may not be improperly employed in commercial rivalry. The phenomena observed are not characteristic of Thomas-Gilchrist metal only, but are also to be met with in Siemens-Martin ingots.

coloured ring. Identical remarks apply to the corresponding girder section shown in photograph 15. In spite of the blow-holes in the ingot, the girder is free from pores and cavities, the blow-holes being completely welded together; and therefore there is no question of the material being a «porous steel». Special attention must be called to this, because had the central portion of the girder section been etched with hydrochloric acid, it would have appeared completely eaten into holes, owing to the local high phosphorus content there. From the ring of moderate sized flow-holes in the ingot lying at the boundary between the central and marginal areas, chains of smaller holes lead vertically to the surface of the ingot. The larger crystals which separated out during solidification in a direction normal to the cooling surface have thrust the gases liberated at the moment of their formation (and in places also certain segregated constituents) against the contiguous surfaces of neighbouring crystals. These rows of minute holes are still distinctly visible in the girder section, forming chains of small welding seams. They are distorted by the rolling process, so that, from the shape they have assumed, weighty conclusions may be drawn as to the manner in which the metal has become distributed during its successive passages through the rolls. Some ideas on the same subject can be obtained by comparing the central and marginal areas in the ingot with those in the girder (see photographs 12 and 14). At the top end of the ingot, the central region occupies about 46 per cent, and the marginal zone about 54 per cent, of the total area. In the web of the girder (also from the upper end of the ingot) the proportion has altered at the expense of the margins; the centre portion occupying about 80 per cent of the space, and the margin only about 20 per cent. In the flanges, on the other hand, the marginal zone is considerably more extensive than the centre. Hence, during rolling, material has been mainly drawn from the margins of the ingot into the flanges of the girder. Since there is a considerable difference between the mechanical properties of the inner and outer regions (some examples of which will be given later), this fact is important to the manufacturer. When a section is etched in the manner already described with cuprammonium chloride, the darker colour assumed by the central portion points to a local concentration of the phosphorus. As the microscope shows, the amount of carbon in the central

area of a section from the upper end of the ingot has also increased somewhat, but not to such an extent as to have any effect on the colouring. The conclusion, drawn from the visual examination are borne out by the following analysis of the ingot:

	Top		Bottom	
	margins	core	margins	core
	%	%	%	%
C . . .	0·06	0·11	0·07	0·07
Mn . .	0·48	0·52	0·46	0·47
P . .	0·07	0·15	0·05	0·08

The figures prove that phosphorus tends to segregate far more than carbon; for whereas at the lower end of the sample no flow of carbon into the central portion is to be detected, a notable concentration of phosphorus has occurred there. The segregation of phosphorus is even greater than analysis shows. The dark spots in the central region of the ingot section represent abnormally high local phosphorus content; and although the author has not hitherto made any determinations of the amount of phosphorus present in the case under description, analogy with similar cases (cf. infra) indicates that the proportion may exceed 0·2 per cent. After rolling, the dark patches reappear as dark stripes, being extended during the operation. The stratification is still more conspicuous in a longitudinal section, because of the greater extension that has taken place. This is evident in photográph 16, which is a representation in its natural size of a girder section

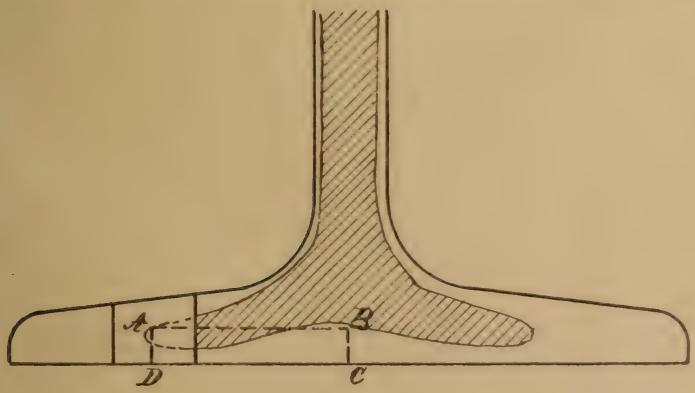

Fig. 3.

different from the one portrayed in photographs 14 and 15. The surface shown is a longitudinal section along the line AB in fig. 3. For completeness, a transverse section on the plane $ABCD$ is given in photograph 17, where only a portion of the central region of fig. 3 is reproduced. From photograph 16 it appears that the long segregation markings are bent out of shape in the vicinity of the punched hole owing to the deformation that has occurred there. The colour round the hole is dark, not because of segregation, but because of deformation consequent upon the punching of the hole. (Compare the remarks previously made about photograph 10 and fig. 2). In bending tests, the girder fractured preferentially in places where the hole fell within a central region, as the crack in photograph 16 shows. Further reference to this matter will be made later on when the mechanical properties of the inner and outer regions come to be described.

The different amount of segregation at the top and bottom of an ingot is shown distinctly in photographs 18 and 19 which represent cross sections of a billet rolled from ingot iron; the former corresponding with the upper end and the latter with the lower end of the original ingot. The dark colour of the centre of photograph 18 indicates that considerable segregation has taken place at the upper extremity, whereas photograph 19 shows, that segregation had occurred in a much smaller degree at the base, The analytical data are:

	Top		Bottom
	margins	core	margins and core
	%	%	%
C	0·08	0·11	0·09
Mn	0·50	0·52	0·50
P	0·080	0·125	0·080

Analysis of large average samples thus shows that there has been practically no segregation at the base of the ingot; but etching tests demonstrate local high phosphorus content in the numerous dark spots developed. The extent of the segregation, however, is much less than at the other extremity.

A characteristic example of high segregation will be found in photograph 20 of a boiler plate section in approximately natural size. The marginal and central areas are clearly to be distinguished,

the dark colour of the latter pointing to high phosphorus content. Etching shows the dark stripes embedded in the central portion to contain a specially large amount of phosphorus, and this is borne out by the annexed analytical data:

	Phosphorus
In the pale outer zone	0·088 %
Along a dark stripe in the inner region	0·203 „
Average over the entire plate	0·168 „

When submitted to low magnification (roughly × 4) as in micrograph 21, the dark stripes exhibit a coarser crystalline structure than their surroundings.

Sulphur as well as phosphorus participates in the segregation. Micrograph 22 (× 123) of the boiler plate that has just been referred to, taken before etching, shows numerous grey particles of sulphides in the immediate neighbourhood of the spots of highest phosphorus content. In order to avoid as far as possible the necessity for a subsequent microscopic investigation when spots of high sulphur content are sought for, the following method of examination has been adopted in the Testing Laboratory. The surface is filed smooth, but need not be polished; on it is laid a piece of silk, which is first moistened with mercuric chloride solution by means of a brush, and then treated with hydrochloric acid. At the places where many particles of sulphides are collected together, sulphuretted hydrogen is evolved, colouring the silk dark, as shown in photograph 23. The segregation of sulphides in the central region of the boiler plate already mentioned, and more particularly along the dark phosphide stripes therein, detected by this test is confirmed by analysis:

	Sulphur
In the outer zone	0·04 %
Along a dark stripe in the inner region	0·16 „
Average over the entire plate	0·10 „

A still more striking instance of sulphur segregation is apparent locally in photograph 24 of a steel plate, where the dark bands indicate segregations of phosphorus. Microscopic examination showed that the amount of entangled sulphide was small in the

outer zone marked I (see fig. 4), but abundant in that marked III, the quantity in zone II being intermediate. On analysis the various zones gave:

	Sulphur
Zone I	0·067—0·069 %
„ II	— —0·201 „
„ III	0·240—0·239 „

Silk tests indicated these differences clearly; and therefore that method of testing may be used instead of a partial analysis, whenever only a qualitative detection of segregated sulphur is required.

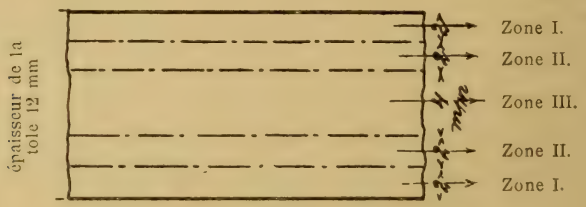

Fig. 4.

Inasmuch as segregations occur in those parts of an ingot that are the last to solidify, oxides which were present in the molten metal or in the ladle, or which were formed during the deoxidation of the metal, are partially carried thither, and are held in place when it becomes solid. Matter entangled in this fashion usually has a bluish colour when viewed under the microscope. In forged or rolled steel the aggregates are elongated in the same direction as the metal; so that the intrusive substances composing them must possess, at the rolling or forging temperature, sufficient malleability to allow of deformation within the mass of iron without losing their continuity. Micrograph 25 (× 123) is a longitudinal section showing three such aggregations within a dark phosphoritic band. The dark spots on the pale ground right and left of the dark band are masses of pearlite in ferrite. Etching with hydrochloric acid would have eaten deeply into the dark band and the matter entangled in it, and would therefore have left no indication of the latter being present; whereas cuprammonium chloride, as micrograph 25 proves, allows all the intrusions to be visible in their original condition. Besides the dark stripes composed of phosphide

segregations, lines of pearlite are frequently to be observed in the longitudinal section of a rolled ingot, as may be seen in micrograph 26 (× 29). The section was etched with alcoholic hydrochloric acid,[1] which does not permit the phosphide segregations to be visible, so that the dark structural constituent there is exclusively pearlite. This peculiar disposition of the pearlite in conjunction with phosphide bands often causes a longitudinal section through rolled or forged steel etched with cuprammonium chloride to exhibit a stratification parallel with the direction of elongation, as is clearly evident in photographs 8 and 9 (longitudinal sections through samples upset when cold). The lines were originally parallel to the axes of the cylinders, but have been bent out of shape by the deformation. Such change in the disposition of the bands forms a very useful indication of the relative amount of deformation and of the positions of strain in the different parts of a specimen.[2] The dark colour in the diagonals of photographs 8 and 9, which has already been stated to be due to deformation, must not be confounded with the markings now being considered, for the latter existed before the deformation took place and have only been diverted from their natural course by it.

Although intrusions of slag, as just mentioned, possess a certain amount of malleability at a forging or rolling heat, the aggregates are ruptured when deformation occurs at ordinary temperatures. The appearance of the masses suggests that of fossil bones broken down by movements in the surrounding rocks. A ruptured intrusion of this kind is visible in micrograph 27 (× 900) which represents an unetched longitudinal section of a fractured test-bar.

In certain circumstances the collections of entangled matter containing oxygen may attain considerable dimensions, as will be evident on inspecting photograph 28. This shows in its natural size a section from a so-called „double“ ingot-plate, rolled in all probability from the upper end of the ingot. Similar phenomena have been met with by Ruhfuss[3], who has found the intrusions

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[1] One cubic centimetre of hydrochloric acid in 100 c. c. of absolute alcohol.

[2] See Zeitschr d. Vereines Deutscher Ingenieure, 1900, Nos. 14 and 16.

[3] „Saigerungen in Flußeisen“ (Segregation in Ingot Iron), by A Ruhfuss. Stahl und Eisen, 1897, page 41.

in cavities of the metal to consist of a grey-green powder, which, on the analysis of three samples, has given the following results:

FeO	MnO	SiO_2	CaO	S	P_2O_5
24.74	63.03	9.16	0.64	0.61	0.227
27.01	59.05	10.18	0.84	0.76	0.316
23.12	71.02	5.01	0.21	—	0.090

These figures (especially the high proportion of manganous oxide) render it very probable that the aggregates of matter containing oxygen are due to a continuation of the deoxidising process within the ingot. Micrograph 29 (× 365) shows that the intrusions of photograph 28 are not homogeneous, but consist of at least three different structural constituents. The oxidised substance thus breaks down into different structural components as it cools.

Comparison between the effect of etching with hydrochloric acid and with cuprammonium chloride. — A few more instances of the different results produced by these two reagents may be recorded, but the author can only refer to such samples as he has been given permission to describe in print. A piece of U-iron (S 671) etched for one minute with cuprammonium chloride is shown in photograph 30, while a setion of the same U-bar etched for 48 hours with hydrochloric acid is represented in photograph 31. Cuprammonium chloride shows the structure of the iron to be very uniform, only a few insignificant patches of high phosphorus content being present. There is no separation into different regions, the only information which can be drawn also from the acid treatment. Photograph 32 is a copper etching, and photograph 33 an acid etching, of an I-girder (S 670). In the former a pale outer zone may be distinguished from an inner core only slightly different in colour, and some darker markings are also to be seen representing notable segregations of high phosphorus content at the junction between the web and the flanges. In the central region of the web, too, a few high phosphorus markings occur, and within them are small intrusions of oxidised matter. (These last are invariably to be found in such positions, and will not be specifically referred to again). The hydrochloric acid (photograph 33) has eaten away the inner region of the metal, thus

exaggerating the differences of the outer and inner regions and making it unable to detect the different behaviour of the different portions of the inner region.

Photograph 34 is the copper etching, and photograph 35 the acid etching, of a rail (S 668); the holes at the base of the former indicating spots whence drillings have been removed for analysis. The rail dates from the early days of rail-manufacturing and is defective by reason of cracks. The perfectly black marks in photograph 34 are actual cavities or cracks. Their disposition is not exactly identical in the two pictures, because the surfaces etched were not contiguous, but belonged to successive sections. There is no distinct separation into different regions in the rail, as shown by the copper etching. But the acid treatment exaggerates this distinction. It is noticeable that the acid has eaten away deep holes (dark spots in photograph 34) at the places of high phosphorus content; so that, without the assistance of the copper etching, there is no means of telling whether these holes represent natural cavities or segregation spots.

The limited utility of hydrochloric acid as an etching reagent is made specially evident in photograph 36 (the copper etching) and photograph 37 (the acid etching) of a steel-rail (S 669). Both sections show a narrow marginal zone with an interior region occupying most of the space. The latter has been seriously eaten away by the acid, whence it might be inferred that the two areas are very different in composition. The copper etching, however, indicates the difference to be much less; a view that is fully supported by the annexed analysis:

	Phosphorus
Marginal zone in the head . . .	0·13 %
Central portion	0·15 %

Actually therefore the phosphorus is distributed throughout the material in a fairly uniform fashion, and a judgment based on the effects of the acid etching would be erroneous. The web of the rail is divided by seams of imperfect welding filled up with slag; in the head the seam is open in places.

It is necessary to remember that unskilful etching with any fluid may lead to erroneous conclusions. If the reagent is not kept in proper motion, currents may be formed in it, and the borders

of the test-surface be attacked more rapidly than the centre. An example of this is given in photograph 38 representing a piece of a steel round bar etched for 24 hours in 2 per cent nitric acid without agitating the fluid, so that currents of different concentration must be formed in it. Actually the metal is quite homogeneous and without the least zonal formation, but irregular etching has caused such to appear A similar phenomenon is evident in photograph 39, where the metal was allowed to rest for 3 minutes in cuprammonium chloride without any disturbance of the liquid. Proof that the zonal appearance is not characteristic of the material lies in the facts that this is not a transverse but a longitudinal section, and that the fallacious paler marginal zone follows the border of the drilled-out hole, the planed-out slot, as well as the periphery of the original bar. Such mistakes will be avoided if the etching reagent is kept constantly in motion; and obviously it is easier to agitate the cuprammonium chloride solution whose action only lasts for a minute than the acid which has to remain in contact with the metallic surface for 24 to 48 hours.

Relationship between the phenomena of an etched surface and the mechanical properties of the metal. — The relationship between the phenomena brought to light by etching a surface with cuprammonium chloride and the mechanical properties of the material have now to be considered. Two particular cases, however, may be put on one side, viz. those where etching demonstrates a local concentration of carbon or the occurrence of a permanent deformation produced when the metal is cold, because the effects of these phenomena upon the mechanical strength of the sample are sufficiently well known. On the other hand, some attention must be devoted to cases of local phosphorus segregation, which is generally accompanied by local concentration of sulphur, and of bodies containing oxygen. As a rule, places where segregations exist have a somewhat higher ultimate strength, and a somewhat lower extension than the rest of the material. The differences, however, are usually not serious, and are seldom large enough to allow a conclusive opinion to be gained as to the effect of the segregations upon the behaviour of the metal — that is true at least of varieties of low carbon steel (boiler-plates, constructional metal). A typical instance of much local segregation

is given in photograph 40, which is an etched transverse section of a connecting-rod screw. On testing and analysing this specimen, the following data were obtained (Table I):

Table I.

Tests of a connecting-rod screw.

(See photograph 40)

	C %	*P* %	*S* %	Yield Point kilos per sq. mm.	Ultimate Strength kilos per sq. mm.	Extension %	Bending No.
Outer zone	0·09	0·050	0·04	25·8	39·6	35·3	3
Inner portion	0·11	0·080	0·08	27·3	43·9	24·9	1

The small increase in ultimate strength (viz. from 39·6 to 43·9 kilos per square millimetre) and the decrease in extensibility (viz. from 35·3 to 24·9 per cent) exhibited by the core of the sample would scarcely lead to the conclusion that the centre should be so much more brittle than the outer zone, as will be shown to be the case later on.

Again, the values for the ultimate strength and the extension (recorded in Table II) of the boiler-plate shown in photograph 20 and micrograph 21 do not bring to light with sufficient clearness the bad qualities of that material:

Table II.

Tests of a brittle boiler-plate.

(See photographs 20 and 21.)

	P %	*S* %		Yield Point kilos per sq. mm.	Ultimate Strength kilos per sq. mm.	Extension mark distance 200 mm. %	Bending No.
Outer zone . . .	0·088	0·04		—	—	—	0—½
Along the dark stripes in the inner portion	0·203	0·16		—	—	—	0
Inner portion . . .	—	—		—	—	—	0—½
Average over the whole section . .	0·168	0·10	longitl.	26·3	42·9	23·8	—
			transv.	[24·6]	42·3	25·4	

This plate was not actually used in practice, for, on applying the cold pressure-test before the boiler was fired up, it fractured along the rivet holes, which had been punched and not drilled.

The plate is rendered unsuitable for employment in boiler construction by its extraordinary brittleness, but no indication of that defect is given by the breaking tests. It is, however, brought clearly to light by the impact bending test applied to a notched sample.

When it is required to carry out this test for brittleness as a consequence of segregation, zonal structure, or improper treatment (e. g. overheating[1]) upon specimens of low carbon steel the following method is adopted in the Testing Laboratory. It has the advantage of extreme simplicity and has already proved most useful: but it is unfitted for application to materials containing much carbon, which should be examined with the aid of a pendulum testing machine.

In view of the common occurence of zonal formation and striation in the material, which renders it necessary to test samples taken from the different parts, each test-bar should be small in size, preferably 60 millimetres long and 4×6 millimetres in cross section (see figure 5).

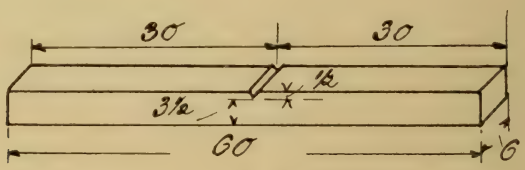

Fig. 5.

On one side a notch 0·5 millimetre deep is cut in it, a tool sharpened to an angle of 60° being employed for the purpose. Each bar is fastened in a vice, as shown in fig. 6, and struck with a hand hammer at the place indicated by the arrow until it is bent to an angle of 90° as there indicated — this is termed the „first bending". The bar is next straightened between the jaws of the vice — the „second bending"; then hammered again — the „third bending" — and so on. The number of bendings it will endure before breaking is called the Bending Number of the bar.

[1] See „The Overheating of Mild Steel", by E. Heyn. J. Iron and Steel Inst., 1902, II. Also „Krankheitserscheinungen in Eisen und Kupfer". (Diseases in Iron and Copper). Zeitschr. des Vereines Deutscher Ingenieure 1902, page 1115; Stahl und Eisen 1902, page 1227.

This Bending Number is a very excellent gauge of the brittleness which occurs in ingot iron poor in carbon. It varies in boiler-plate from 0 to 4. The latter figure is only given by the best materials in the best conditions. Zero represents great brittleness due either to bad material or improper heat-treatment. The sample referred to in table I gives a Bending Number of 3 in the outer zone, but of 1 only in the inner portion; hence the latter is very considerably more brittle than the metal of the outer zone. The utility of the same bending test is still more clearly demonstrated by the metal of table II, which, by reason of its mechanical tests, would pass the 1902 Würzburg rules for «Boiler-Shell-Plate, II» and the corresponding 1905 rules for «Boiler-

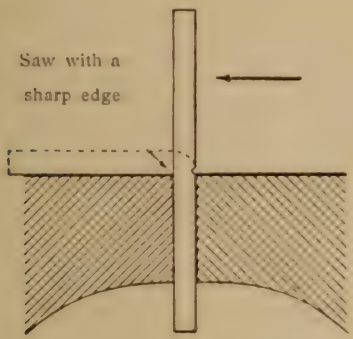

Fig. 6

Shell-Plate». The bending test, however, gives numbers ranging from 0 to $^1/_2$, and shows the metal to be extraordinarily brittle. Zero indicates that the sample cracks at the first blow of the hammer.

As a result of numerous investigations, it may be stated that the local segregations of phosphorus rendered visible by the etching test are regularly accompanied by a reduction in the Bending Number, i. e. by an increase in brittleness; so that some opinion as to the properties of a metal may be derived from a study of the etched section. Naturally, it is advisable to seek confirmatory evidence in the bending test.

The cause of certain phenomena in bending and breaking tests. — The etching test throws light upon certain phenomena met with in breaking and bending experiments. For

instance, if the phosphorus segregations exist in masses of considerable size, they become visible on the broken surface of a test-piece (compare photograph 41) by spots being paler than their surroundings. A longitudinal section through the fractured surface after etching with cuprammonium chloride is represented in photograph 42. The places where pale markings show in the fracture are the extremities of dark bands which lie parallel to the rolling direction of the metal (and in this case also parallel to the axis of the bar). The colour of these bands points to their containing much phosphorus, as is borne out by analysis:

	Phosphorus
In the bands (or markings) . . .	0·43%
Elsewhere	0·08%

The high phosphorus content of the material within the bands causes it to possess low extensibility, and it cannot therefore participate in the greater extensibility of the neighbouring metal without fracturing. This explains the multitude of small cracks existing in the bands as indicated by fig. 7. If the preparation

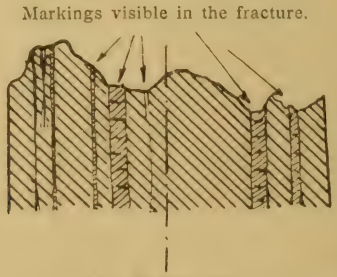

Fig. 7.

of the test-bar causes these lines of substance rich in phosphorus to extend to the surface, the well known hard-veins[1] are produced. The cause of their formation has hitherto not been understood, but the etching test makes the reason evident. A view of such hard-veins is given in photograph 43.

[1] «Materialienkunde für den Maschinenbau», page 81. By S. A. Martens. Also «Mitteilungen aus den Königlichen Versuchsanstalten», Berlin, 1890, supplementary volume II.

The brittleness of materials made from ingot iron with noteworthy phosphorus segregations is brought into prominence when the metal is exposed to shocks after being injured (notched). In boiler construction such injuries are caused by punching the rivet holes. As the right hand portion of photograph 20 shows, the lines of high phosphorus content are sharply bent in the direction of the punching (cf. fig. 8). Sometimes the microscope proves the metal to

Fig. 8.

be fractured, so that from the surface of the hole fine cracks *r r* penetrate into the material, especially into such portions as are is most susceptible to injury. Plates of this kind are prone to fail suddenly along the riveting lines when, as is inevitable, they are exposed to shocks. When a rod having a structure such as is represented by photograph 40 is turned down with a sharp angle as at *a* in fig. 9, a notch is made in the inner region of the metal which

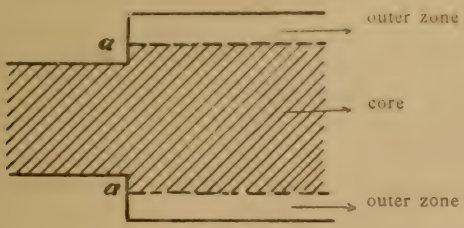

Fig. 9.

is so sensitive to injury; and therefore in consequence of a shock it will break along *a a*. If the cut does not penetrate into the interior portion, but is limited to the less sensitive outer zone, the same rod is much less likely to fracture. Nevertheless, it must be remembered that an indentation is present; and therefore it is advisable to investigate with the aid of the bending test the degree of resisting power against sudden stresses possessed by the metal when it has suffered an injury of the kind described.

The case of a screw-bolt is similar, for there exist on it rows of indentations; and the object must be viewed with suspicion whenever the bottom of the thread enters into a core which shows a tendency to brittleness owing to segregation phenomena.

Sampling for chemical analysis. — The etching test also teaches several lessons as to the manner in which a sample should be taken for analytical purposes. As a general rule, the fewest errors are introduced by taking a cut from over the entire section; but it is judicious first to apply the etching test in order to detect any heterogeneity. Too little attention is still bestowed in many laboratories upon the effect which the method of sampling may have on the trustworthiness of the results. Samples are often taken with the drill or on the lathe in an unsuitable fashion. Very many analytical differences which are commonly attributed to the methods employed are actually due to improper sampling; and no doubt, much of the disagreement amongst chemists as to the methods that should be adopted springs from the same cause. Several incorrect methods of sampling may be quoted. If, for example, holes are drilled to different depths in a plate as shown in Fig. 10; and if there has been a concentration of phosphorus

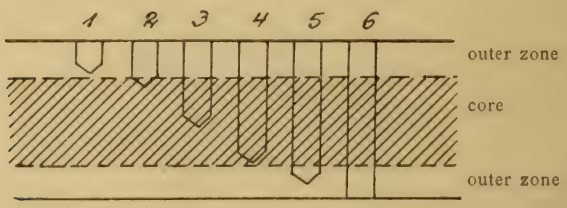

Fig. 10.

in the central region of the metal, the drillings from each hole will give different figures for the phosphorus content. The correct proportion of phosphorus can only be ascertained from the drillings of the hole (No. 6) that passes right through the plate. If the drillings from the different holes are sent to different chemists for analysis, the various phosphorus determinations must of necessity be different whenever the composition of the outer zone is not the same as that of the interior of the mass. The same remark applies to the determination of the sulphur, and also, though to

a smaller degree, to that of other foreign substances present. When the plate represented in photograph 24 and fig. 4 was sampled in the manner shown by fig. 10, the drillings of the different holes (Nos. 1 to 5) gave figures for the sulphur ranging from 0·068 to 0·17 per cent.

Flat steel bars with a very masked core cannot be correctly sampled by taking the drillings from a hole driven right through it. The drillings obtained from the two holes *a* and *b* in fig. 11

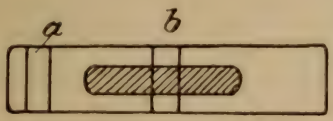

Fig. 11.

are of different composition. In such a case the metal should be planed over its whole section. When a round bar such as is shown in photograph 40 is drilled at I with one hole (cf. fig. 12)

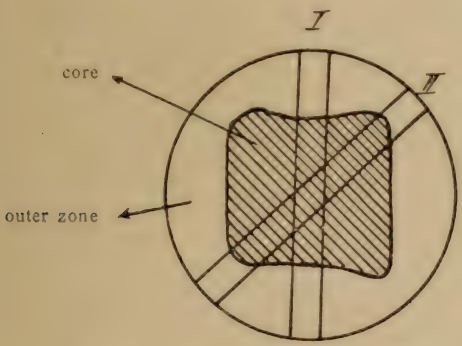

Fig. 12.

the borings do not give its average composition correctly as is the case with the planings from a cut taken off its whole transverse area. Even the position of the holes I and II produces some difference in the results Very serious errors are made if a bar is sampled by taking a cut from it in the lathe, for the first turnings will contain less phosphorus than those which follow. — In certain instances analytical differences crop up even when the material has been sampled properly. Suppose a round steel bar, which

is to be turned up by the engineer who purchases it, is tested for phosphorus in its rough state by the manufacturer, the sample being taken correctly in the form of a planing over its whole transverse area, and suppose the proportion of phosphorus is found to be *a* per cent. When the purchaser has turned it down to a smaller diameter and inserted it in any machine or structual work, a premature fracture of the bar decides him to make his own determination of the phosphorus; and he does so in a sample obtained, in the same correct manner as before, from a transverse cut over the entire section of the bar—his result being *b* per cent. If, now, the bar contained spots of abnormally high phosphorus content in its core, *b* will naturally come out higher than *a*.

Distinction between wrought and ingot iron. — The etching test affords considerable assistance in ascertaining whether a particular material is wrought iron or ingot iron, whereas examination of a fracture often leads to incorrect conclusions in that respect. It is characteristic of wrought iron to be built up of different varieties of metal each having its own structure. An example is given by photograph 41 which represents a flat-bar of wrought-iron. The bands of different structures are distributed irregularly, as might be expected to ensue from the rolling of a composite mass. The dark patches denoting high phosphorus content in certain places are also to be observed, and their significance is proved by analysis:

	Phosphorus
Present in a dark stripe	0·16%
Average of the whole bar . . .	0·067%

The transverse section of a piece of ingot iron may certainly exhibit structural differences in different places, as is evident from a study of photograph 40. But the distribution of these differences obeys a certain rule, which depends on the phenomena of congelation and the treatment that follows it. A further distinction between wrought and ingot iron lies in the nature of the intrusions containing oxygen. In wrought iron the intrusions of hammer-slag are generally distributed in masses of considerable size, having either an uniformly dark colour, or being composed of two differently

coloured bodies, as is shown in micrograph 45. Ingot iron usually contains intrusions of slag of microscopic dimensions, the most important features of which have already been discussed.

Welding methods. — Etching with cuprammonium chloride shows clearly whether welding seams exist in a material, and in what manner the welding has been performed. Photograph 46 represents an oblique lap weld; photograph 47 shows a but welded, and photograph 48 a „patent welded" tube. It is also easy to tell if a tube is seamless or has been welded. Much insight can be gained into the amount of care exercised during welding if the etching process is followed by microscopic examination.

The structure of fractured surfaces. — The surface of a fracture is often examined in order to judge the condition of a material; and much may be learnt thereby if all the factors of the case are taken into consideration. In the routine of steel manufacture, the appearance of a fracture is deemed important. Nevertheless, as a general rule, trustworthy conclusions can only be drawn in this fashion provided the investigator knows the condition under which the material has been produced, the manner in which it has been heated and handled mechanically afterwards, and the way in which the fracture has been made — otherwise there is great opportunity for error. The appearance of a fracture does not depend only on the nature of the material, but is partly due to the fracturing process. This point need not be laboured, although a few instances may be described where the non-homogeneous structure of a fracture might lead to the opinion that the material itself had been structurally heterogeneous. In photograph 49 is shown the fractured surface of a bar, in which the great difference in colour and granulation between the centre and the margins might well cause the suspicion that a large flaw in the metal had been brought to light. Actually, however, the material is homogeneous, and is proved to be so by the view given in photograph 50 of a section perpendicular to the fracture. The differences in the appearance of the fracture are due to the fact that it is jagged at the part *a b* (fig. 13), but comparatively smooth at *a c* and *b d*. Photograph 51 represents the fractures of some test-bars prepared from a particular steel. They exhibit dark markings which might be imagined to come from some other material. That however, is not

the case; the structure is perfectly homogeneous. The peculiarities arise from causes similar to those that produce cupping.[1] In photograph 52 will be seen the fracture of a tyre made of special steel. The stratification might easily induce even an experienced investigator to think that the different striæ represented differences in

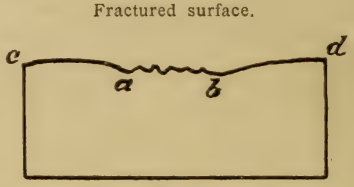

Fig. 13.

structure or irregular chemical composition. This again is not the truth, for the whole effect depends on the phenomena of fracturing. The material is composed of fibres whose major axes lie nearly perpendicularly to the plane of fracture, as is evident from an inspection of photograph 53 and fig. 14. Hence, where the fracture takes

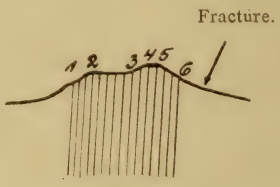

Fig. 14.

place obliquely through the component fibres (as at 1—2, 3—4, and 5—6) it has a different appearance from that which it possesses where it cuts them at right angles (2—3, 4—5).

Presence of old flaws. — One of the most difficult problems ever submitted to the investigator is to decide whether the failure of a constructional material has been brought about through the existence of an «old flaw», because only in exceptional cases does he see the fractured surfaces in their original condition before they are coated with rust. Since the etching test fails to answer such a question, its discussion does not properly fall within the scope of the present report; but inasmuch as the zonal

[1] Materialienkunde, page 79. By A. Martens.

structure of ingot iron is so far concerned in the matter that it deserves to be kept in mind, a few words may be devoted to one particular point which has frequently led to mistakes in the past. The inner region of ingot iron rusts in air more easily than does the outer zone, so that if the fractured material possesses a strongly marked core, that portion of the metal often becomes visible without the aid of any etching process soon after the failure has occurred. It would therefore be a serious error of judgment to conclude from this phenomenon that the rusty part of a fracture represented an «old flaw», while the remaining bright portion was that which broke afterwards; nor is it necessary to elaborate any theories as to the causation of internal flaws which accurately follow the configuration of the inner region of the metal.

Crystallisation during the cooling of ingot iron. — The etching test often performs useful service when the crystallisation occurring during the solidification of the ingot has to be studied. This crystallisation may be termed the «first crystallisation», in order to distinguish it from the „second crystallisation" which proceeds in the solid state during the transformations that occur in the iron at a temperature above 700° C. but below a higher point that varies somewhat. The latter is the cause of the true fine structure visible under the microscope when the metal is lightly etched. As a rule, the new structure it produces entirely masks that due to the first crystallisation, which is only rendered evident by powerful etching with cuprammonium chloride or other reagents. Surfaces of least resisting power are usually formed during the first crystallisation, and these influence the nature of the fracture and the mechanical strength of the material far more seriously than does the fine secondary structure. An excellent example of this is shown in photograph 54 representing the fracture of an ingot iron casting. When etched with cuprammonium chloride, as in photograph 55, the coarse fibrous structure of the first crystallisation is brought clearly to light. Heating has a certain effect upon the gradual change in the crystallisation; but even heating for half an hour to from 900° to 935° C. is by no means sufficient to alter it entirely, as photograph 56 shows. The effect of forging and rolling upon the alteration of the first crystallisation is apparent from photographs 57 to 59. The first represents a portion of

a transverse section of the ingot-casting, which contained 0·39 percent of carbon. The upper edge of the picture, corresponding with the surface of the ingot, has a coarse fibrous structure perpendicular to the surface; but towards the lowest part of the photograph, which corresponds with the centre of the ingot, the fibrous nature gradually disappears. Photograph 58 is a transverse section through a bloom forged from the ingot after cutting it longitudinally into two equal portions. The fibrous structure perpendicular to the three surfaces of the ingot is still distinct; but about the bottom of the picture (which coincides with a diameter of the ingot) it vanishes, and the original interlaced formation [„tannenbaumförmige Kristallisation", or fircone structure] takes its place. Photograph 59 shows a round rod rolled from the bloom; and the previous remarks are applicable here also, although the signs of the first crystallisation are still less evident. If the area of the test-piece is yet further reduced, the traces of the first crystallisation vanish altogether. The three photographs 57 to 59 explain why forging and rolling, and the reduction in transverse area which accompanies those operations, improve the quality of the metal. Their sole object is as, far as possible, to intertwine the coarse crystals that were formed during the solidification of the metal, and so to reduce the number of surfaces of least resistance lying between and in the crystalline aggregates, which are besides contaminated by segregations of impurities and occluded gases. The amount of reduction in area to which an ingot is subjected during its conversion into a finished piece of constructional metal is therefore of great importance, as practice has shown to be the case. This is also true of the material in the inner core of ingots in which much segregation is present, as will be shown by the following instance. Severel bars for tensile tests were prepared out of the distinctly marked core-zone in the bulb and in the web of a bulb-girder, as the annexed table indicates: The former exhibited a considerably lower elongation than the latter owing to the fact that the bulb has undergone less reduction of area during rolling than the web.

	Ultimate Strength kilos per sq. mm	Extension $l = 5{\cdot}65\sqrt{f}$	Reduction in area %
Inner Portion of Head . .	60·8	11·8	10
Inner Portion of Web . .	61·7	23·0	41·5

Etching and microscopic examination occasionally afford an insight into the heat-treatment to which ingot iron has been subjected, as the following example shows. A boiler-plate had bulged when in service, and the question arose whether the bulging was caused by faults of the material or by a local rise of temperature in the boiler. A sample of the plate taken as far as possible from the bulge is shown in its natural size in photograph 60 after being etched with cuprammonium chloride. The structure is fine in grain, like that presented by rolled plates. At the spot which bulged, however, the plate exhibits the structure indicated by photograph 61. Here the ferrite particles are large and the structure is coarsely crystalline. Such a change in structure is only to be produced at high temperatures and therefore the problem was solved. It remained to be decided whether the heating had produced any „overheating" of the metal, for this may also produce large grains of ferrite. But when „overheating" occurs the metal is rendered more brittle, and consequently the Bending Number falls. Tests carried out in the manner already described showed this not to be the case, for the coarsely crystalline metal of the bulged spot gave a Bending Number of 3, while the fine grained metal from a distance gave 2. Hence „overheating" had not taken place, and the local rise in temperature had not only not rendered the metal more brittle, but had actually improved it.

A section through a plate which has assumed a coarsely crystalline structure as the result of overheating is shown in photograph 62. The Bending Number has fallen to 0 or $\frac{1}{2}$, and the material has become extremely brittle. By raising it to a temperature of 900° C. the traces of overheating are removed, and the Bending Number becomes normal again.

These examples show that a material does not necessarily suffer from brittleness because its etched surface exhibits a coarsely grained structure; in such cases the etching test only indicates a need for further examination.

In the foregoing report the lessons to be learnt from a visual examination of an etched metallic surface are dealt with only so far as they apply to malleable iron in its unhardened condition. The utility of the test has been sketched briefly, for the author desired to present a general survey of the results to be obtained, rather than to enter upon an exhaustive treatment thereof. Examination by the unassisted eye after a surface has been etched gives most valuable information as to the character of quenched high carbon steel, and to this branch of the subject the author hopes to return in a future communication. The applicability of the etching test is by no means limited to varieties of iron, for it can be employed with excellent results in the study of copper and its alloys. In this case, however, it is preferable to use a 1 : 12 solution of cuprammonium chloride mixed with excess of ammonia as the active reagent.

Charlottenburg, 23rd January, 1906.

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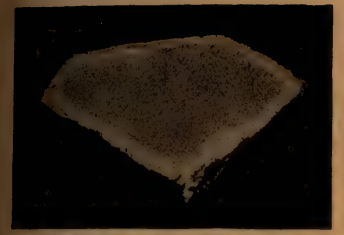

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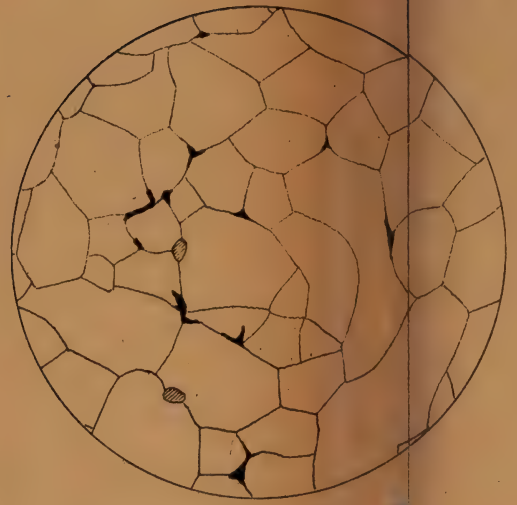

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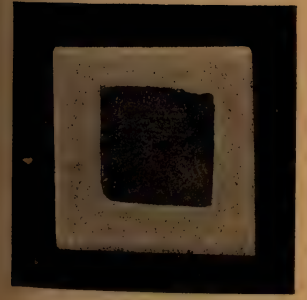

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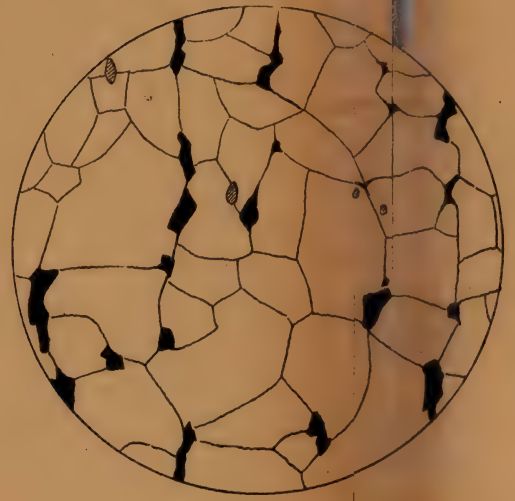

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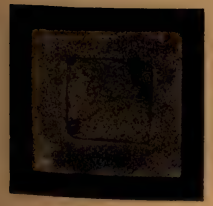

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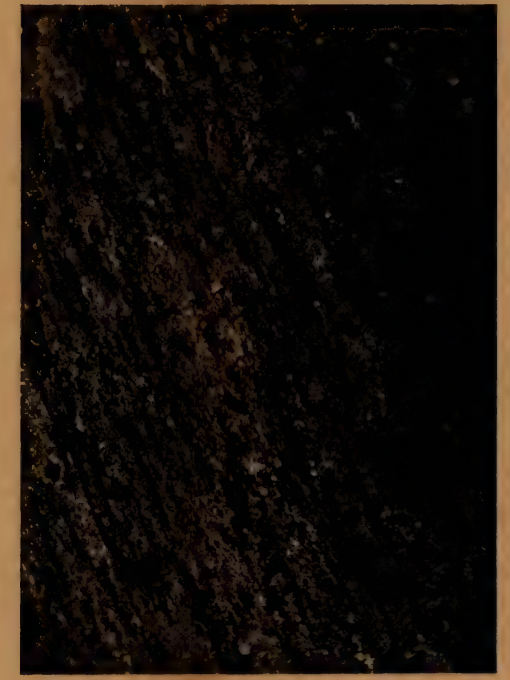

11

Top-end

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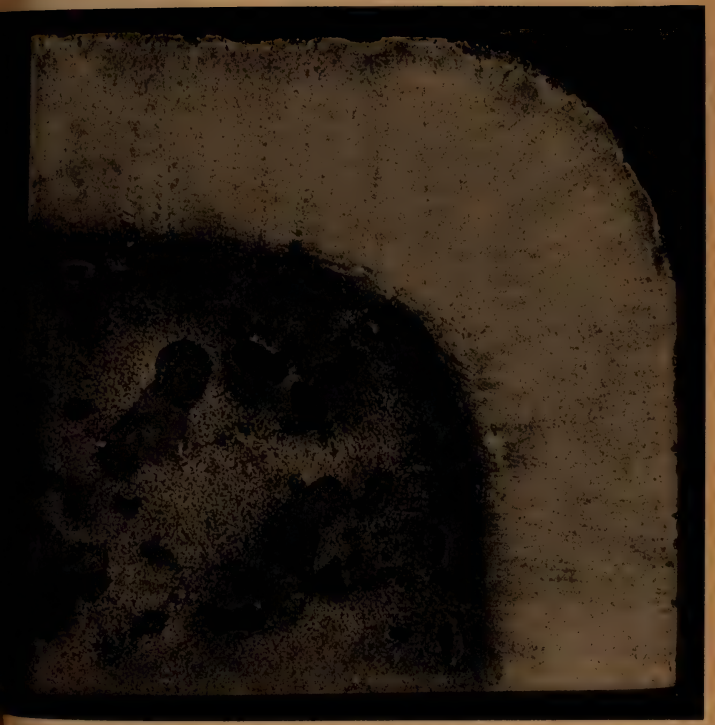
Cast-ingot.

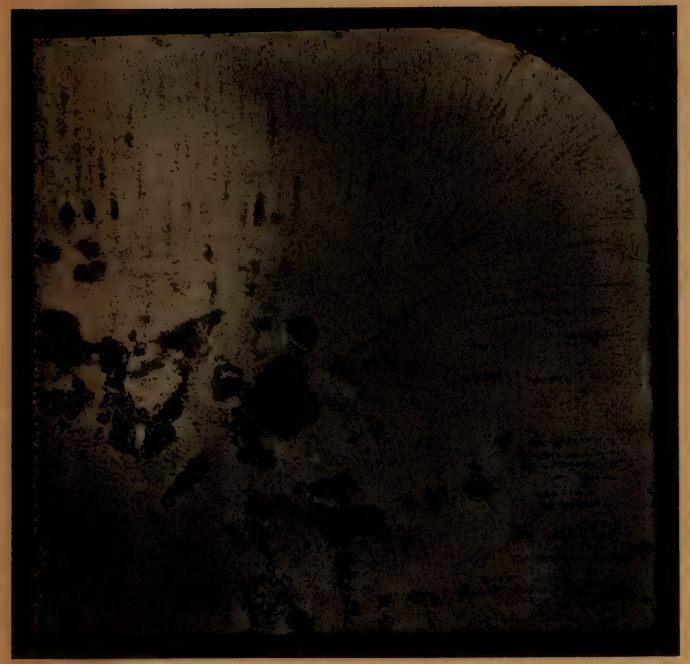
Top-end



12

One quarter of the ingot in approximately natural size.

Bottom-end



13

One quarter of the ingot approximately × 0.88.

Girder rolled from the ingot shown.

Top-end

14

Bottom-end

15

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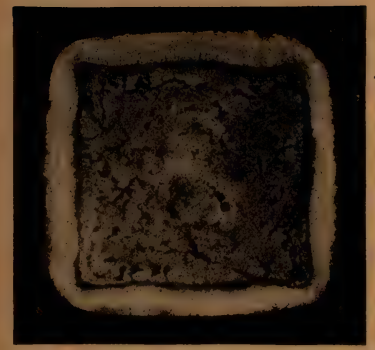

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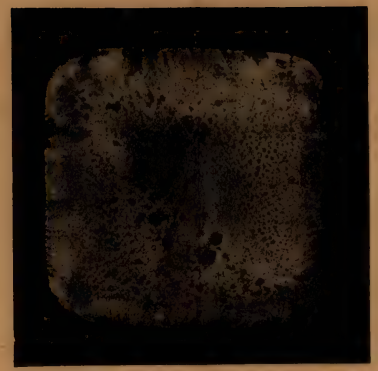

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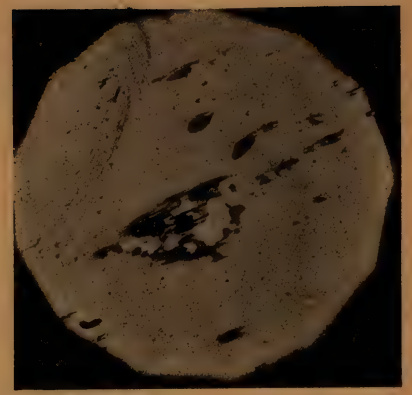

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× 123

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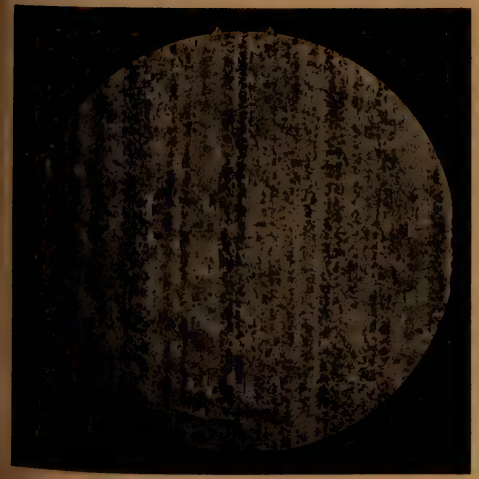

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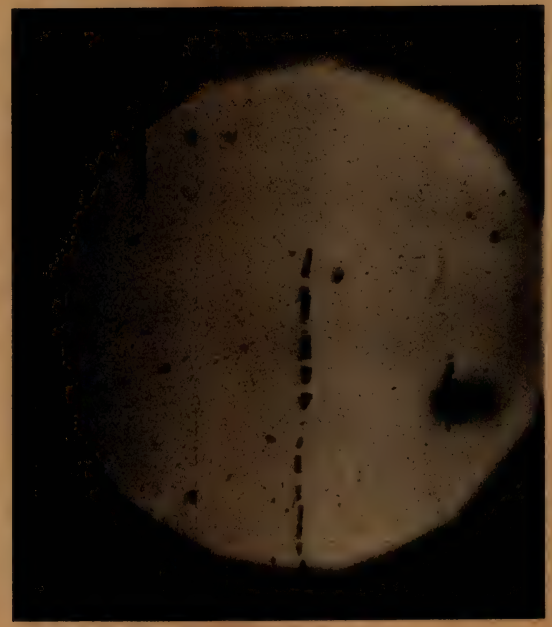

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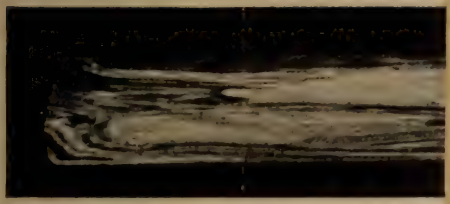

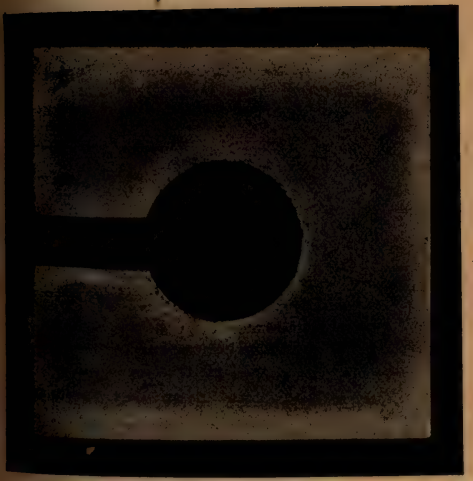

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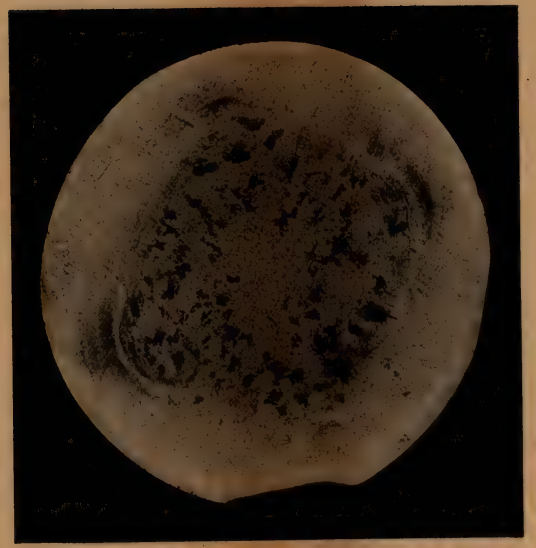

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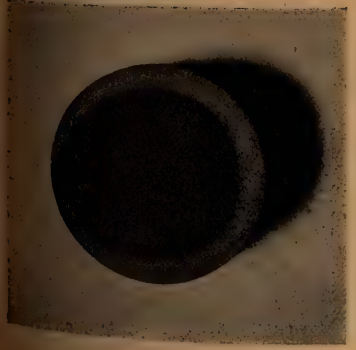

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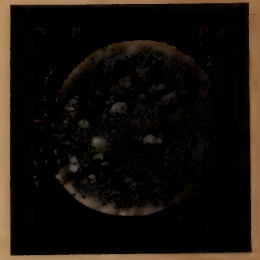

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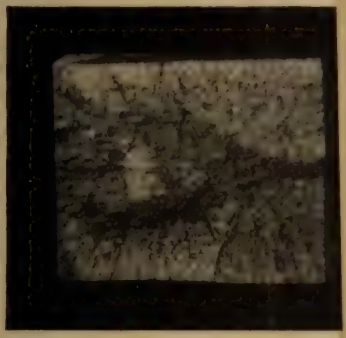
Plane of fracture.

ection of striation.

53



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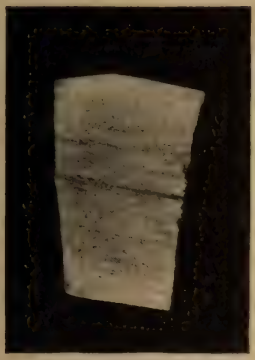

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Plane of fracture.

Direction of striation.

53

54

49

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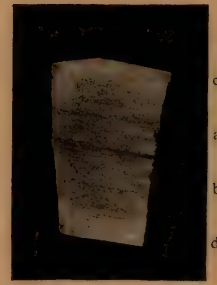

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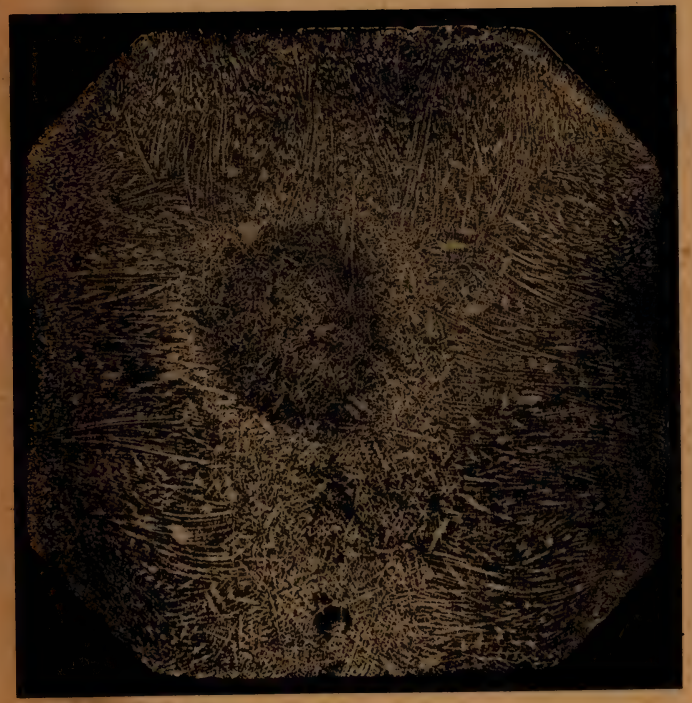
59

Round bar 15 mm in diameter,

Surface of the ingot.

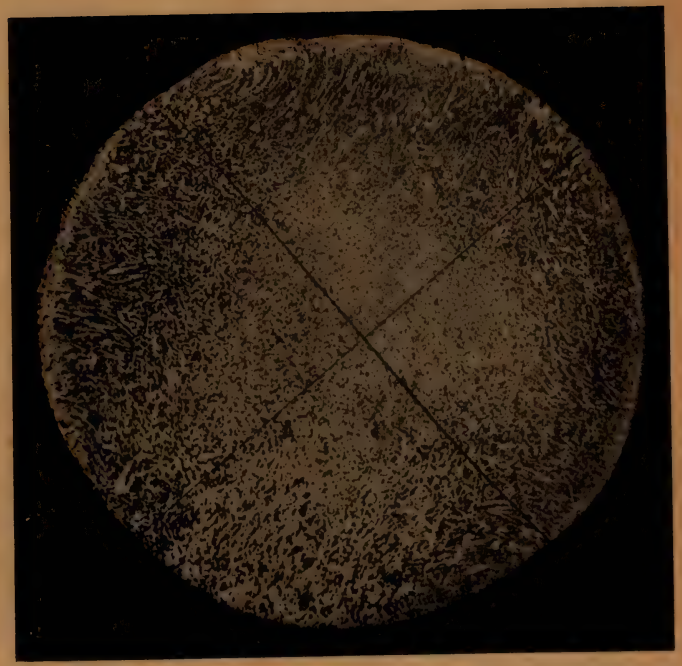
57

Transverse section of the ingot 60 × 100 mm.
Only a part of the section is shown.



58

Bloom about 40 × 40 mm in transverse section, forged after bisecting the ingot longitudinally.



59

Round bar 15 mm in diameter, rolled from the bloom.

60

61

62

4 e.

INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 4.

METHODS FOR THE EXAMINATION OF WELDING AND WELDABILITY.

BY
PROFESSOR **REINHOLD KROHN,**
GEHEIMER REGIERUNGSRAT IN DANZIG.
(TRANSLATED FROM THE GERMAN ORIGINAL).

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Methods for the examination of welding and weldability.

By Professor **Reinhold Krohn**, Geheimer Regierungsrat in Danzig.

Translated from the German original.

The problem submitted to Commission 4 of the International Association for testing materials relates to researches on »methods for the examination of welding and weldibility«.

This problem was taken over by the Zürich Congress in 1895 and referred to a Commission at a meeting of the Directors in Vienna on the 26/27 April 1896.

The former chairman Professor H. Gollner, of Prague, who has since died, in a circular, issued in 1895, which contained fundamental determinations for the solution of the problem, tried to induce the members of the Commission at that time to proceed with this research.

The following gentlemen replied to the circular: Professor Belelubski, Chief Engineer, St. Petersburg; Chief Engineer Eckerman, Hamburg and Mr. Knaudt, Factory Proprietory, Essen, the two first named gentlemen gave their sanction generally to the proposals set forth in the circular, whilst Mr. Knaudt was of a different opinion. The other members of the Commission did not favour the discussion of the questions with their consideration.

Even a further attempt by Mr. Gollner, to advance the work of the Commission, failed.

After the death of the former chairman, the undersigned was requested by the President of the International Association to take the chair of Committee 4 and to make suitable proposals.

For the latter purpose I conferred first, with several local experts particularly experienced in the special field in which the researches of the Committee lay. From interviews I had with these gentlemen I became convinced that the problems submitted to the Committee could scarcely be satisfactorily settled. The Committee was dissolved at the meeting of the Directors on the 1st and 2nd March 1902. Below I give my report, and since I am called upon to report on the question, I beg to propose the motion, «that the Congress withdraw this problem from the list and make a note of my report».

It is extremely difficult to formulate a standard-test for estimating the weldability of a material; such a test should exclude any element of chance and in addition to defining the shape and mode of working of the test-piece, should also state the manner, duration and degree of heating to which the iron should be subjected before and during the process of welding.

Precautions should also be taken to ensure the proper observance of these conditions at the experimental welding.

In the case of the standard-test, welding by hand is naturally excluded, the welding would have to be done by mechanical pressure or blows. Provisions should also be made to ensure a distinctly definite pressure, or distinct energy of the blow. It seems particularly difficult to prescribe and carry out measures for the purpose of ensuring that the material during the welding is maintained at a certain definite temperature, and several members of the Commission have already seriously doubted the possibility of so doing.

Even if it were possible to prescribe and carry out a standard-test which would exclude any personal factor, such a test would scarcely be of practical value.

In carrying out the welding process with the different materials used for welding namely wrought-iron, ingot-iron and steel, they are not treated in the same way in order to obtain a good weld.

Even by restricting the standard-test to one of the aforesaid materials, say ingot-iron, a uniform welding-test would not positively prove the possibility of welding since the kind of weld depends in a critical manner on the properties of the iron. To obtain a good weld, ingot-iron must be subjected to different

treatment, according as it has been produced by the acid or basic process either in the open hearth furnace or the converter, and also according to its strength and chemical composition. The temperature and duration of heating, the energy of the blows, and the kind of flux to be used varies with the different sorts of iron; in each individual case exhaustive tests are required to determine the treatment most suitable for producing a weld.

It is well known that, if works accustomed to always welding the same sort of iron, have for some reason or other to use a material different to that used ordinarily, it is necessary at first to make several tests and trials before obtaining a good weld with the new material.

Even slight variations in the properties of the iron necessitate an alteration in the performance of the welding, and for this reason as is shown by experience, boiler-makers, chain-makers etc. who undertake welding on a large scale, always prefer the same material, from the same source and obtained from the same raw materials, because only this course guarantees a good and safe result in the welding. It is therefore impossible to succeed if only one standard method of testing be adopted (for one class of material e. g. that grouped under the term „ingot-iron") as the nature of the test would have to vary according to the hardness and the source of origin of the material.

And further if these difficulties could be overcome, the standard welding-test would not be of practical value.

Such a test should prove whether certain material can be more or less effectively welded in order to decide whether the material may be successfully employed for welding purposes. As a matter of fact, this end cannot be achieved. Naturally welding carried out in regular factory-service is not done in exactly the same manner as when welding for a standard-test. As, however, as stated above, the quality of the welding depends in a great measure on the way in which the tests are made, a good result from the standard welding-test does not in any way guarantee the reliability of the welds made with the same material in large works.

For the examination of the welds standard-tests of course can be devised. In this case the directions and the carrying out of the tests would be much simpler than in the case of the

standard-test with regard to the welding properties of the materials. But even tests on the quality of the weld are of little practical value. If a test-piece is taken from a welded part, and the quality of the welding examined, the result of this examination gives no correct idea of the quality of the remaining part of the welding. The quality of the welding depends from point to point on the skill, the care and the reliability of the workman entrusted with the work; for the same weld may be good in one place and defective in another. The conditions under which other tests of materials are made are essentially more favourable. It may, for instance, be readily assumed that the material of one and the same charge of ingot-iron shows in its entire mass a certain homogeneity, so that tests made on several bars, selected from the charge, may lead to a satisfactory conclusion as to the properties of the whole material derived from the same charge as far as its behaviour in the mills is concerned.

The examination of a single weld however can only be used very guardedly in forming an opinion as to the quality of other welds, and consequently very little value can be ascribed to standard welding tests.

It must accordingly be concluded that the problems submitted to Committee 4 of the International Association, as far as they admit a solution generally, are but of little value, while, that part of the problem relating to the ascertainment of the welding properties of materials, and for the solution of which the establishment of directions for a «Standard weld» was proposed, cannot be effectively dealt with.

It seems, therefore, advisable to strike out problem 4 from the list of the Association.

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 25.

TO ESTABLISH UNIFORM METHODS OF TESTING CAST IRON AND FINISHED CASTINGS.

REPORT PRESENTED
BY THE CHAIRMAN OF COMMISSION 35
DR. RICHARD MOLDENKE,
SECRETARY OF THE AMERICAN FOUNDRYMEN'S ASSOCIATION, NEW YORK.

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APR 25 1911
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To establish uniform methods of testing cast iron and finished castings.

(Proposed at the Budapest Congress 1901.)

Report presented by the Chairman of Commission 35 Dr. **Richard Moldenke,** Secretary of the American Foundrymen's Association; New York.

The Commission appointed for the solution of problem 25 consisted of following members:

Chairman:

Moldenke, Richard, Dr., Secretary of the American Foundrymen's Association, New York N. Y.

Members:

Belgium: Doat, ingénieur civil; 388, rue des Vennes, Liége.

Danemark: Krog, Fr. A., Direktor der Aktiengesellschaft Frederiksberg, Eisengiesserei und Maschinenfabrik; Kopenhagen F.

Germany: Joly, Hubert; Wittenberg.

Wüst, Dr., Professor der technischen Hochschule; Aachen.

Jüngst, C., Geheimer Bergrat; Charlottenburg, Kurfürstendamm 214 p.

England: Stead, J. E., Laboratory and Essay-Office; Middlesborough.

France: Charpy, G., Dr., Docteur-és-sciences, ingénieur aux usines, St. Jacques; Montluçon (Allier).

Hollande: Muysken, J., Direktor der Nederl. Fabrik von Werktuigen en Spoorweg material; Amsterdam.

Italy: Fadda, S., chef de la traction et du matériel des chemins de fer de la Méditerranée; Turin.

Norway: Ihlen, N. C., Ingenieur, Besitzer von Strömmen Mek. Werksätte, Strömmen per Christiania.

Austria: Jüptner, H. Freiherr v., k. k. Professor an der technischen Hochschule; Wien IV., Karlsplatz.

Lenz, Edgar v., Eisen- und Stahlwerksbesitzer; Traisen N.-Ö.

Russia: Bureau des forges; St. Pétersbourg, Potschtomskaja 13.

Switzerland: Sulzer-Großmann, A., Maschinenfabrikbesitzer; Winterthur.

United States of America: Outerbridge Al. E. Pr. Metallurgist 1600, Hamilton Street, Philadelphia, Penna.

Sauveur, Alb., Professor, Manager of Boston Testing Laboratories; Cambridge, Mass.

West Thomas, D. Foundry Expert, Sharpville Pa. Harvard University.

The peculiar nature of the material we have to deal with, the comparatively recent knowledge of the constitution of cast iron acquired by the producer rather than the consumer, and the question of commercial ethics this naturally brings about; all combined, have tended to make the subject of testing cast iron, and the co-related writing of proper specifications, a most difficult one to bring to a point of satisfactory solution. Nevertheless much work has been done in recent years to bring the testing of cast iron up to date, and the tendencies this has led to should be studied closely with a view of international agreement on the lines of best results to foundry and testing laboratory combined.

Up to the time of taking hold of this matter by our technical societies, what little testing of cast iron there was carried out, was confined to the cross-breaking of long square test bars, the supporting of a given weight on the part of which, or the actual destruction with higher loads than the ones specified, gave the necessary information to the buyer. When a foundryman did care to know what his iron was worth for strength, he would take shorter bars of square or round section and break them transversely, and thus gain an approximate knowledge of what he was doing.

With such an imperfect system in vogue in Europe and America, very little can yet be expected from our first effort to

prepare specifications for the testing of cast iron, until thoroughly proven in the commercial part of the industry. To judge from the work of the two countries which have published specifications for testing, namely the United States, and Germany, it will be seen that Germany has been fit to retain the old long bars in some measure and elaborate tests with them, while America has cut loose from the old methods and developed a system which would take into account the heat treatment of the metal, as it were, and yet free the foundry industry from troublesome requirements as much as possible. The founder, in his daily occupation, is left to test his metal as he wishes, and an „arbitration bar" so called, cast under conditions as nearly perfect as can be made without unessential complications, stands between maker and buyer when a dispute arises and the quality of the metal be questioned.

As there are only two series of specifications to be presented to the International Association for testing materials, they will be taken up in the essential features herewith:

In Germany there have been adopted specifications for Machinery Castings, Columns, and for Pipe. In the United States of America, for General castings, Locomotive Cylinders, Malleable Castings, Car Wheels, Pipe, and Pig Iron for Foundry purposes. Specifications for Coke are now being prepared.

It will be noted that these products of foundry and furnace selected for specification, are the result of conditions peculiar to the respective countries. Germany does not make cast iron car wheels. On the other hand America is getting away from cast iron columns as fast as possible for important structures. Germany specified three test bars, each a standard for its corresponding divison of castings as to their thickness. Thus thin castings of all kinds come under one test bar with various requirements of strength. Medium sized castings have another standard test bar, and similarly heavy castings the third. In American practice but one bar is provided for the general classes of castings, and the strength under test is so adjusted that it covers what is considered first class metal for thin, medium, and for heavy castings.

On studying the methods used for the preparation of the test bars in both countries, it will be noted that a commendable attempt is made in each case to get as near the ideal conditions under which a bar should be cast, as is possible. Whatever may be the ultimate

trend of methods of testing cas iron, we can congratulate ourselves on the fact that the industry is not hampered by the accumulation of work done by previous generations which it is desired to retain even though the basis upon which this was made is incorrect.

A comparison of the two series of specifications appended to this report*) will show that the specifications adopted in Germany and in the United States are not very far apart, in fact could be changed but little without destroying their usefulness in their respective countries. After all, a very essential point, so far as the science of testing is concerned, is that they agree in the particular directions which may be considered basic. They can safely be let stand on their merits until time will bring with it improvements and possibly such changes in the industrial markets of the world that a closer agreement between specifications can be brought about.

Regarding the methods of testing themselves, this much may be said. The peculiar nature of cast iron precludes the use of the tensile test for commercial purposes. In fact only with the most accurately adjusted testing machines can reliable tensile tests be made. Hence the German specifications have omitted this test altogether, while in America the test appears practically under protest. The transverse test seems to be the one more generally accepted as adapted to the work in hand, and with careful observation, a fair measure of the value of the material is obtained.

Impact tests have not so far proven their value in the foundry industry, nor have punching, shearing, nor making any tests disclosing new avenues of research, appeared on the horizon. It is to be hoped, however, that research will continue and that the future will give us further aid in our endeavor to improve the products of the foundry.

Watchung, N. J., July 31st, 1906.

*) Vorschriften für die Lieferung von Gußeisen, aufgestellt vom Verein deutscher Eisengießer.

American Society for Testing Materials. Standard specifications for gray iron castings, for cast-iron car wheels, for cast-iron pipe and special castings, for locomotive cylinders, for malleable castings, for foundry pig iron.

These specifications appended to the report were not reprinted.

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INTERNATIONAL ASSOCIATION FOR TESTING MATERIALS.

BRUSSELS CONGRESS 1906.

REPORT ON PROBLEM 26.

REPORT ON IMPACT-TESTS ON NOTCHED BARS.

BY **ED. SAUVAGE**, INGÉNIEUR EN CHEF DES MINES, ENGINEER-IN-CHIEF TO THE WESTERN RAILWAY CO. OF FRANCE.

TRANSLATED BY CH. CAPITO, LONDON.

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Report on Impact-Tests on Notched Bars

by **Ed. Sauvage.**

(Translated from the French Original by Ch. Capito, London.)

Introduction. — The Budapest Congress (1901) of the International Association for Testing Materials passed the following resolution: that in addition to the usual tests which are at present made when metals are tested for acceptance, and also for the purpose of study, there should be carried out as often as possible, impact-tests on notched bars, with chisel, with impression of balls, in order to find out how the results from the various methods correspond with eachother and to point out exactly the numerical data applicable to the various qualities of metals.

At a general meeting of the Congress, it was agreed to submit again to Sub-Committee 22 the question relating to impact fragility-tests, with the mission to define the experimental conditions to be adopted in carrying out these tests, and to make beforehand the necessary experiments for arriving at this result.

In a letter dated June 12th 1903, the President of the International Association expressed his desire to entrust the study of impact-tests on notched bars to the author of this report. To facilitate this study, the following questions were put to a great number of engineers and manufacturers interested in the matter:

1. Are impact-tests on notched bars made in your country?

2. Are these tests made in laboratories as studies on scientific principles?

3. Or, on the outher hand, are they made in practice when accepting metals, either as a complement to other specified requirements, or as obligatory tests?

4. Kindly give a complete and detailed description of the methods of impact-tests for experimental purpose, or as actually used?

5. What is the shape, and what are the dimensions of the notches you use?

6. Do you possess any comparative results of impact-tests and tension-tests?

7. Have you found that very slight differences in the shape of the notches, such as those which result from a trifling wear of the tools, have a great influence on the result of the tests?

8. Can you indicate the conditions, to be applied in impact-tests, such as the height of the drop and the weight of the hammer, which permit, in the case of certain qualities of steel, of separating the metal which should be accepted, from those which do not show sufficient, guaranty?

9. The author will be very glad to have your opinion on the question of impact-tests on notched bars, and also any remarks you may have to make on this subject.

The author has received a large number of answers containing documents of the highest importance, notably from:

Mr. C. Haberkalt, Chief-Engineer to the Ministry for the Interior, Vienna.

Professor B. Kirsch, Technische Hochschule, Vienna.

Mr. Wilhelm Hauser, Engineer to the Ministry for Railways, Vienna.

Mr. Rayl, Director of the Kaiser Ferdinands-Nordbahn, Vienna.

The Board of the works of the Staatseisenbahn-Gesellschaft, Vienna.

The Board of the Rima-Murány-Salgó-Tarján Works at Budapest, with two collections of experimental results and a large number of test-diagrams.

Professor Rejtö, Polytechnikum, Budapest.

Mr. Ch. B. Dudley, President of the American Association for testing materials, with reports and a letter from Mr. S. Bent-Russell, Engineer, a collection of specifications and a bibliographical notice on impact-tests

Professor W. C. Unwin, London.

Mr. J. E. Stead, Head of the Laboratory and Assay Office, Middlesborough.

Mr. R. A. Hadfield, Director of Hadfield Steel Foundry Co., Sheffield.

Professor H. J. Hannover, Copenhagen.

Mr. S. A. Lund, Chief-Engineer, Christiania.

Mr Schroeder van der Kolk, Chief-Engineer of Railways (Holland).

MM. C. J. Snyders and P. A. M. Hack-stroh, Engineers, the Hague.

Prof. Mesnager, Paris, with an extensive collection of results from tests.

Mr. Solacroup, Engineer in Chief of the Paris-Orleans Railway, with a collection of tables containing results of tests.

The author sincerely thanks these gentlemen who have thus given him substantial aid in the task imposed upon him by the Committee of the Association.

Professor Rudeloff, of Charlottenburg, has published an interesting memoir in „Stahl und Eisen" (April 1 and 15, 1902), on tests of iron and steel by means of notched bars. A translation into French of this memoir by M. Breuil appeared in „La Revue de Mécanique" of August 1902 (p. 145). In his memoire M. Rudeloff mentions numerous works on this question.

In France, the Society for the Encouragement of National Industry printed in 1904 under title of „Contribution à l'étude de la fragilité dans les fers et les aciers" (Contribution to the study of fragility in irons and steels.) a collection of memoirs relating to impact-tests and to the study of fragility, some of which were of a recent date, and others of an earlier date and had already been published This collection contains memoirs by Messrs. Considère, Brustlein, A. le Chatelier, Ast et Barba. Codron, Auscher, Frémont, Osmond, Charpy, Vanderheym, Huillier, Guiliery, Leblant, Mesnager, Fain, de Fréminville. M. H. le Chatelier, in an introduction, sums up the state of the subject.

An interesting review on the subject of impact-tests appeared in the Bulletin number 5 (October 1899) of the American Section of the International Association for Testing Materials.

A study of impact-tests by Commandant D. Francisco Ceron y Cueron of the Spanish Artillery, was published in 1902 in the „Memorial de Artilleria".

Messrs. Yarrow have made impact-tests of Nickel Steel an account of which is given in „Engineering“ of April 18th 1902, page 513. These tests are also mentioned in the Proceedings of the Institution of Mechanical Engineers (Novm. 1904, page 1185) The same number of these proceedings contains an important paper by Messrs. A. E. Seaton and A. Jude on this kind of test; a resumé of this paper will be found further on. In the Revue de Mécanique (Jan. 1905, page 52) will be found an abstract translation of Messrs. Seaton and A. Jude's memoirs.

In a paper entitled „The relations between the effects of stresses slowly applied and of stresses suddenly applied in the case of iron and steel presented to the Iron and Steel Institute by P. Breuil and published in the Institute's Journal (1904, No. 1), the author comes to the conclusion that the effects due to the slow actions are similar to those due to the sudden actions. For this reason, he does not believe that tests on notched bars are destined to substitute tensile tests. This paper is reproduced in French in the Bulletin du laboratoire d'essais mécaniques, physiques, chimiques et de machines du Conservatoire national des Arts et Métiers (t. I, 1903—1904).

In the first year's issue (1904) of the „Revue de Métallurgie“ is published an interesting study by Messrs. F. Osmond, Ch. Frémont and G. Cartaud, on „the modes of deformation and rupture of iron and mild steel“ (p. 11 and 198). This review also contains several memoirs which have been reprinted in the collection cited above.

The printed minutes of the meetings held by the Union of French and Belgian Members of the International Association for testing structural materials, contain numerous communications on the subject from Messrs. Chartiée, Guillery, Charpy, Mesnager, Guillet, Pérot.

Apparatus. For impact-tests on bars of small dimensions, several appliances are used, which measure the energy absorbed by rupture, with an approximation corresponding to the degree of precision which is obtained in practical tests carried out on metals which are never perfectly homogeneous. The spring hammers of Barba and Frémont, the pendulum hammer, and Guillery's rotary hammer may be specially mentioned.

The spring hammer used by M. Barba and M. Leblant for the Company of the Chemins de fer de l'Est (Eastern Railway of France) is arranged so as to rebound on springs, the energy remaining in the hammer after fracture being measured by the height to which the hammer rebounds on striking a spring. This apparatus is described with illustrations in the report of MM. Ast and Barba to the Budapest Congress.

The Frémont hammer has been described many times, notably in the „Bulletin de la Société d'Encouragement" (September 1901, p. 372.)

The energy remaining in the hammer, after rupture of the test bar, is measured by the compression of a spring. This apparatus is usually constructed with a fall of 4 m (4.374 yards) and with hammers weighing 10 and 15 kilos. (10 kilos is 22 lbs. 3/4 oz and 15 kilos is 33 lbs. 1 oz.)

The pendulum hammer has been employed notably by Mr. Bent Russell, Mr. Charpy and Professor W. C. Unwin. It consists of a mass suspended by rods which allow it to swing like a pendulum. The pendulum is turned a fixed amount from its vertical position and is then allowed to fall: the rupture of the test bar takes place when the pendulum attains its vertical position, and the remaining energy is calculated from the height to which the hammer swings after impact. The mass of the suspension rods is not a negligeable quantity compared with that of the hammer, and a considerable error would be made, if the energy be calculated from the weight and heights traversed by the hammer alone. Still it is easy to correct this error, and it may be done after the blow, if necessary. This hammer has been described by Mr. Bent Russell in the „Transactions of the American Society of Civil Engineers" (June 1898 p. 237) and by M. Charpy in his note on the testing of metals by impact bending-tests of notched bars, published in 1901 for the Budapest Congress.

Mr. Bent Russell has described a modification of the pendulum hammer (Transactions of the American Society of Civil Engineers, June 1900, p. 6) constructed to make tensile tests by impact. The rectangular test-bar is notched in the middle on two parallel faces; it has two heads, one of which is fixed in the machine, and the other is fixed to a piece that is struck by the pendulum hammer.

Guillery's rotary hammer is of a more recent date than the preceding ones and has the advantage of occupying less space. It has been described in the „Revue de Métallurgie" (Aug. 1904, p. 405). It consists of a small steel fly-wheel carrying on its rim a knife intended to break the test-bars. The energy absorbed by the test-bar is calculated from the variation in speed of the fly-wheel at the moment of rupture. This is measured by the aid of a tachometer in the form of a small centrifugal pump forcing back water into a graduated vertical tube. Other tachometers may be used. The test-bar is placed on a movable anvil, which keeps it back from the fly-wheel at the commencement of the trial. A simple mechanical device allows it to approach the fly-wheel at the desired moment. This device consists of a catch which is struck by the knife when pushed by the aid of a lever. The catch then releases a spring which brings the anvil against the fly-wheel, and at the next passage of the knife, rupture takes place. The apparatus is completely enclosed in a metal casing.

Two different models have been constructed by which energy of 60 and of 200 kilogram-meters can be produced respectively at a speed of about 300 revolutions per minute.

Comparative experiments have been made by MM. H. Le Chatelier and Mesnager, by means of Guillery's rotary hammer and Frémont's hammer. They were carried out on test-bars 1 square cm in section and with rounded notches 2 mm deep. A tuning fork apparatus was mounted on the Guillery hammer, to register the speed and to check the indication of the centrifugal pump tachometer. The energies of rupture per square cm in kilogram-meters are as follows:

	Guillery's hammer		Frémont's hammer
	Tachometer pump	Tuning fork apparatus.	
	15	13·4	13·4
	20	17	14·9
	17·5	15·3	14·9
	15	15·3	16·1
	13·6	15·7	13·4
	13·6	15·7	12·7
Averages:	15·7	15·3	14·7

The consistency of the results may be regarded as very satisfactory.

M. Pérot has published in the „Revue de Métallurgie“ (1904, p. 287) an original method for studying impact phenomena: the hammer carries a photographic plate, on which are registered, by the action of conveniently directed light-rays, partly the displacements of the anvil-block, mounted on springs, due to the effect of the impact, and partly the marks corresponding to regular intervals of a very small fraction of a second. This method permits of a minute analysis of the short lasting phenomena which accompany the impact.

Test-Methods. The test bar is usually of rectangular shape and nicked on the opposite face to that which receives the blow of the hammer. It is placed on two supports and struck in the centre in such a way as to be broken by a single stroke. The weight of the hammer and the height of the fall remain the same for each size of test-bar.

The nick is produced simply by a saw-cut, or in a tool and then trimming it by a perfectly smooth impression of a wedgeshaped steel, or by drilling a cylindrical groove and clearing off the part left between the face of the bar and the groove.

The ingenious method of M. Barba, which consists of preparing a series of notches on a long bar, successively clamped to the right of the notches, and struck by the hammer with gradually decreasing heights, is rather difficult to apply in practice under ordinary circumstances.

M. Chartier, inspector of the P. L M Railway, reported to the French members of the International Association on May 7th, 1902, with regard to the method used, when accepting rolled steel to be used for bridges and metal framework; the tests according to this method are prescribed in the specification of the P. L. M. Ry. for supplies of iron and steel for above named structures, as is shown in the following abstract of this specification.

Impact-Tests.

Impact-tests are to be carried out on test-pieces cut cold from the sheets, plates and sections which are sent to be accepted. These test-pieces must never be annealed.

Whatever may be the thickness of the sheets, plates and sections, the test-pieces shall invariably be 70 mm long and 30 mm wide. Two semi-circular grooves of 15 mm diameter shall be bored in the middle of the length of the bar.

The thickness of the test-piece shall be the same as that of the bar to be tested.

The two semi-circular grooves shall be bored. The four sides of the test-piece shall all be cut by means of a rectangular punch 70 × 30 mm.

The impact-tests shall be carried out crosswise. When the bars are not wide enough to allow of the test-pieces being selected in this manner, i. e. when the width is less than 70 mm, then the impact-tests shall be carried out lengthwise, but only in that case.

Ten per cent of the bars sent to be accepted shall be tested by impact. The Company's Inspectors have the right to increase this proportion for certain classes of bars, and to reduce it, by way of compensation, for certain other classes.

With a view to impact-tests, all bars shall be rolled with an excess length of 80 mm, whatever may be the direction in which the test is made.

Test. The test-piece, such as defined above, shall be placed on a steel die the rectangular groove of which is to be 50 mm long and 35 mm wide. The weight of the anvil-block shall be at least 800 kg

The hammer, whose weight shall vary according to the thickness of the test-piece, as mentioned below, shall be furnished, at its lower part, with a steel percuteur, the shape and dimensions of which will be shown on a sketch.

The test-pieces shall be able to support, without breaking, the shock from a hammer falling through a height of 4 m in the direction of their section of fracture. The adjoining table indicates the weights of the hammer for each particular case.

All test-pieces which are cracked but not broken shall be considered to have proved satisfactory in the test.

If all the test-pieces taken from bars belonging to the same charge are broken, then the whole charge shall be rejected. But in this case, the maker may, if he thinks fit, test all the bars which have not previously been tested. Those bars which are found to stand the test shall be accepted.

If the test-pieces selected from bars of the same charge give inconsistent results, then all the other bars of the same charge shall be tested.

The test-pieces, each 70 mm long, 30 mm wide, and of the full thickness of the bar from which they are cut, are provided with two lateral semi-circular notches, 15 mm in diameter, made with a drill or in the milling machine. The width between the notches is thus reduced to 15 mm. This test-piece resting on two

Crosswise Tests of sheets		Crosswise Tests of plates of width = 0m,070		Crosswise Tests of angles & sections of width — 0m,070		Lengthwise Tests of plates, angles and sections of width \ 0m,070	
Thickness of bars	Weight of Hammer	Thickness of bars	Weight of Hammer	Thickness of bars	Weight of Hammer	Thickness of bars	Weight of Hammer
8 mm	6,00 kg	8 mm	5,00 kg	8 mm	6,00 kg	8 mm	8,00 kg
9 „	7,00 „	9 „	6,00 „	9 „	7,00 „	9 „	9,00 „
10 „	8,50 „	10 „	7,50 „	10 „	8,00 „	10 „	10,50 „
11 „	10,00 „	11 „	9,00 „	11 „	9,00 „	11 „	12,00 „
12 „	11,50 „	12 „	10,50 „	12 „	10,00 „	12 „	13,50 „
13 „	13,00 „	13 „	12,00 „	13 „	11,00 „	13 „	15,50 „
14 „		14 „	14,00 „			14 „	17,50 „
15 „		15 „	16,50 „			15 „	20,00 „

supports 50 mm apart, receives on one of its plane faces, between the two lateral notches, the blow of a hammer falling from a height of four metres. The weight of the hammer varies with the thickness of the piece, from 5 to $16^1/_2$ kg for thicknesses from 8 to 15 mm.

Mr. Bent Russell has made tests by longitudinal blows on notched test-pieces, with the apparatus first described.

In addition to measuring the energy expended in fracturing the test-piece, the impact-test gives other indications which may be recorded with advantage, such as the angle of bending after rupture and the deformation of the transverse section.

Mr. C. Haberkalt, Chief-Engineer of building-constructions (Ober-Baurat) to the Ministry of the Interior in Vienna, has communicated the following observations on testing metals for use in public bridges.

Impact-tests are not tried, but bending tests of notched bars are made according to a specification published in 1892. The test is made with bars from 50 to 80 mm wide, the thickness being that of the sheet or section from which they have been selected. They are cut perpendicularly to the line of rolling and have a notch traversing one tenth of their thickness. The bars are bent cold over a circular piece, of which the radius (in the case of Martin steel) is one fifth of the thickness. The angle of bending before rupture should be at least 90 deg. in the case of a steel having a tensile strength of 45 kg per sq. mm and 150 deg. for a strength of 35 kg per sq. mm.

Mr. C. Haberkalt adds that it is important in this test to observe the effect of the shape of the notch; the eventual use of the impact-test for notched bars requires that light should be thrown on this point.

The same bending tests on notched bars are used in Austria for railway bridge steels, as stated by Mr. Wm. Hauser.

Professor B. Kirsch of the Technological Museum of Industry, Vienna, believes that it is impossible to lay down general rules for the impact-test on notched bars of different qualities of metal. This opinion is the result of his researches on comparing compression-tests and impact-tests (usually without notches).

Mr. William Hauser, Engineer of building construction (Baurat) to the Ministry of Railways, Vienna, does not think that the impact-test on notched bars can be easily made practical for testing materials to be accepted. The difficulty seems to him still greater in the case of steel for bridges, than with thicker bars such as rails, axles, and tyres.

The Board of the Rima Murány-Salgó-Tarján works (Budapest), have sent an important collection of diagrams and tables of tensile tests on ordinary bars and on notched bars according to the method of Professor Rejtő. This company however does not make impact-tests on notched bars. Professor Rejtő has appended some remarks to his table of tests.

Mr. Rayl, Director of the Kaiser Ferdinands-Nordbahn, the Board of the State Railway Co's works at Budapest, as well as Mr. Lund of Christiania, have stated that they have neither documents nor observations to present on the subject of tests with notched bars.

Mr. Bent Russell has published, in the transactions of the American Society of Civil Engineers (June 1898, p. 237, and June 1900, p. 1) his tests with transverse blows as well as with tension by impact. Various materials were tested, in particular, bricks, wood, and cast iron. The bars of wood which were tested had triangular nicks. The conclusions of Mr. Russell are favourable to impact-tests on notched bars, but he has not found any connection between the results of ordinary tensile tests and those of impact-tests. He takes moreover the view that in impact-tests on notched bars, the form of the notch should be guided by the conditions under which the metal is to be used. Thus a rounded nick is best for pieces which have to be pierced with rivet or bolt holes, while an angular notch would be more suitable for pieces where abrupt changes in section take place.

Professor Unwin, can as yet give no conclusions on the subject of impact-tests which he has made with a pendulum hammer.

Mr. J. E. Stead as yet knows of no application in Great Britain of impact-tests on nicked bars when accepting delivery of metals. He announces that he intends to make some experiments with test pieces notched according to the Charpy method. The simplicity of these tests and of the necessary apparatus is of great interest.

Mr. R. A. Hadfield also announces the installation of Frémont's hammers at Sheffield.

According to Mr. Hannover's reply, impact-tests on notched bars are not used in Denmark, but only the well-known impact tests on whole pieces such as axles and tyres, and sometimes they are notched so as to cause them to break.

Mr. Schroeder van der Kolk, Chief-Engineer of railways in Holland, has carried out tests with the Frémont hammer, principally on steel taken from bridges. The object was to ascertain whether Bessemer steel in certain parts ought to be replaced by the softer Martin steel. The tests were made in the laboratory of MM. Koning and Bienfait, Amsterdam, and about 200 test-pieces were tried.

Mr. van der Kolk thinks that the influence of the greater or less rounding of the angles, produced in nicking with the saw, is negligible, all the ruptures having occurred at the bottom of the nick.

The test-pieces are classified as follows, according to number of kilogrammeters expended in breaking them.

Work in kilogrammeters per sq. cm.	Number of bars.	Work in kilogrammeters per sq. cm.	Number of bars.
0 to 1	19	13 to 14	1
1 „ 2	16	14 „ 15	1
2 „ 3	29	16 „ 17	1
3 „ 4	32	17 „ 18	3
4 „ 5	31	18 „ 19	2
5 „ 6	20	19 „ 20	2
6 „ 7	12	20 „ 21	4
7 „ 8	10	21 „ 22	4
8 „ 9	9	22 „ 23	5
9 „ 10	1	25 „ 26	1
10 „ 11	5	26 „ 40	7
11 „ 12	1		

The pieces of Bessemer steel which gave the worst results in these tests have been replaced by Martin steel.

The effect of annealing has also been tried on a charge of 15 test-pieces, 3 were annealed at 750 deg. C for 3 hours, 3 others from 850 to 880 deg. for 4 hours, 3 at 950 deg. for 2 hours and finally 3 at 1050 deg. for 2 hours. The influence of the annealing upon the work done in breaking the bar was inappreciable and microscopic examination showed that it had caused very little structural change.

With test-pieces of Martin steel, the energy spent in breaking varied greatly according as to whether they were cut with or across the direction of rolling.

Mr. van der Kolk remarks that the indications of the Frémont hammer may be erroneous if the test-piece undergoes much bending during rupture. He prefers moreover to measure the kinetic energy of the hammer after each rupture by the method of rebound.

Mr. van der Kolk has found no connection between the results of tensile tests and impact tests; as is shown by the table he has prepared which is given below.

Energy of rupture by shock on notched bars in kilogrammeters per sq. cm. .	11	10	8	3	1	0	28	21	19	18	18	17	11	3	3	2	1
Breaking load with tensile tests in kgs per sq. mm .	44	44	44	44	44	44	44	43	40	43	39	42	42	43	41	43	43
Elongation per cent . . .	24	21	26	25	28	28	25	27	30	28	27	28	26	26	26	26	29

Mr. van der Kolk has investigated the relation between the energy of rupture by impact and the deformation of the transversal section of the broken test-pieces. With this in view, he measured after rupture the width of the test-piece on the side a which had been struck, and also the width of the opposite side, viz. along the notch b; he then compared the differences $b-a$ with the corresponding values of the energy of rupture The table below, which sums up this comparison, shows that a certain ratio exists between these two quantities. These figures only apply to test-pieces which fracture along a plane and not to those which require a great energy of rupture.

In summing up, Mr. van der Kolk is of the opinion that one cannot yet fix in a rational manner the lower limits of the energy of rupture acquired for each quality of metal. He thinks that this might be determined by studying pieces which have given good results in actual service.

In their reply MM. Snyders & Hackstroh, engineers, show that impact-tests on notched bars are carried out in Holland by military engineers, but only for scientific purposes, and not with the view of being used as an acceptance test of metals. Their conclusions are unfavourable to this kind of test. They have published a memoir on the subject in Dutch, and the conclusions are as follows:

»Static bending tests, whether on notched or unnotched bars, give results analogous to those found by impact-tests, whether on notched or unnotched bars. Hence it is evident that impact-tests convey no information not given by the static bending tests.

The results of impact-tests on bars notched on the side opposite to the one which is struck are useless for ascertaining the fragility of steels.«

Mr. Mesnager has carried out in the physical and mechanical testing laboratory of the Ecole Nationale des Ponts et Chaussées, in Paris, a very extended series of impact-tests on notched bars. Several thousands of test-pieces have been broken. The tests were carried out for comparison with Charpy's oscillating hammer and with Frémont's hammer. Mr. Mesnager has made a special report, the conclusions of which are as follows:

1. The results vary generally less in the case of large bars with cylindrical notches (Charpy type) than with small bars (Frémont type).

2. The relation between the number of kilogram-meters per sq. cm of the fractured section and the angles of deformation in degrees may be expressed by the following formulae:

Small test-pieces $K = 0{\cdot}375\ D$
Large „ „ $K = 1 + 0{\cdot}58\ D'$

3. Hence we can thus substitute the magnitude of the angle of rupture for the amount of energy absorbed in rupture.

4. The relation between the resistance to tension in kg. per sq. mm., and the angles of rupture and also the kilogram-meters per sq. cm. may be expressed approximately by the following formulae:

Small test-pieces $R + 2{\cdot}66\ D = 95$ $R + 7{\cdot}1\ k = 95$
Large „ „ $R + 1{\cdot}72\ D' = 87$ $R' + 3\ k' = 90$

5. A metal with blow-holes seems to require more energy of rupture than a sound metal.

6. Large test pieces of homogeneous metals give results of remarkable regularity.

7. The notch terminating in a cylinder of 4 mm in diameter and made with a drill, and the notch made in the milling machine, give practically the same results.

8. Small variations in the span and height of the test-piece make very little difference in the result of the tests, if the section of fracture remains the same. The width of the notch on the other hand modifies them considerably.

Number of the bars, classified according to the energy of rupture in kilogram-meters per sq. cm), and to the differences b — a (in deci-millimeters), shown in the first column.

	Energy of rupture:													
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	kilogram-meters per sq. cm														
Bessemer Steel															
b—a = 0 deci-millimeters	1	1		1											
1 „ „	2	1	2	2	2	1									
2 „ „	2		1	3	2	1									
3 „ „			2	2	1										
4 „ „		1	1	1	1	2	1								
5 „ „			2	1	1	4	1								
6 „ „					1	1	1		1						
7 „ „								1							
8 „ „						1		4							
9 „ „									1						
10 „ „								1							
11 „ „															
12 „ „									1						
13 „ „					1										
14 „ „									1						
Mild Steel (Martin and Thomas)															
b—a = 0 deci-millimeters	2	1	1												
1 „ „	3	3	3	4											
2 „ „	3	2	2	5	2	1									
3 „ „	4	3	4	4	2	1									
4 „ „	2	2		2			1								
5 „ „		1	2	1	3	2									
6 „ „				4	5	2									
7 „ „				2	2	1	1								
8 „ „					4	1					1				
9 „ „					2	1	2								
10 „ „					2	2	1	1	1			1			
11 „ „				1				1	1						
12 „ „						2									
13 „ „							3	1	3		1				1
14 „ „							1		1		1				
15 „ „															
16 „ „									1						
17 „ „											1				
18 „ „												1			

Mr. Guillet has drawn the following conclusions from a series of impact-tests which he made with a view of studying the fragility of steels and more particularly that of mild steels, after heat treatment and particularly after superficial cementation:

1. The steel delivered, in spite of being recommended, was in no sense homogeneous, and unfortunately this is generally the case.

2. Proper quenching improves all these steels considerably.

3. The divergencies obtained in the first tests were due not to the method employed but to the heterogeneity of the metal.

4. In order to estimate the homogeneity of a metal, it is necessary to try as many test-pieces as possible by impact-test, and consequently to use test-pieces with as small a section as possible (without exaggerating the smallness of the test-piece itseft).

Taking test-pieces of a section thicker than that adopted by Mr. Frémont, we obtain averages, but we do not know the differences.

To test a bar through all its thickness, does not show the degree of homogeneity of the metal.

A very heterogeneous bar, tested as a whole, may give a result which is considered satisfactory, and in actual use it may break after a longer or shorter service, on account of its want of homogeneity.

Mr Solacroup, Chief Engineer of the Paris and Orleans Railway Co., has made a series of tests with a hammer similar to that of the Eastern Railway Co., on test-pieces with triangular notches. The results were compared with those obtained with the Charpy hammer and the Frémont hammer, on test-pieces of the same origin.

The diagrams which represent these experiments show a certain relation between the energy of rupture by impact and the results of tensile tests. The results with the Charpy hammer and that of the Eastern Railway Co., agree fairly well, but the results with the Frémont hammer did not seem to agree with either of them.

Mr. Guillery, manager of the works of the Société Française de constructions mécaniques, at Denain, has made comparative tests of the Charpy and Frémont methods, on steel plates for locomotive frame plates. As the two methods gave widely different

results, a very homogeneous piece of a plate was chosen and cut into 15 bars of the Charpy type with notches perpendicular to the rough faces of the plate, and 15 others with notches parallel to these faces. After each test, each bar gave 2 to 4 test-pieces of the Frémont type.

The Charpy test-pieces with notches perpendicular to the rough faces gave values of energy of rupture per sq. cm varying from 32 to 23·3 kgm; those in the other direction gave values varying from 38 to 49 kgm. With the Frémont test-pieces, the results kept between 30 and 40 kgm per sq. cm. For this reason, Mr. Guillery concluded that the Frémont process gives the most correct results.[1])

Mr. Le Blant has carried out a series of tests in the mechanical testing laboratory of the Chemins de fer de l'Est, with notches of different forms, as follows:

1. Very acute angle, milled and corrected by compression by means of a finishing tool.
2. With a sharp saw 2 mm thick.
3. With a slightly worn saw, 2 mm thick.
4. With a round toothed saw 2 mm thick.
5. With a sharp saw 1 mm thick.
6. With a slightly worn saw, 1 mm thick.
7. With a round toothed saw 1 mm thick.

Mr. Leblant concludes from his tests:

That in the case of very great fragility, the shape of the notch is of little importance.

That the heterogeneity of the test-pieces completely obscures the differences due to differently shaped notches, as well as the influence of the wear of the saws or cutters.

That the acute angled notch, the most severe one, gives results more easily comparable, than other notches.

Among the most remarkable results of the impact tests, Mr. Dumas, consulting engineer to the Commentry-Fourchambault Co., cites chrome-nickel-steels, the notched bars of this material could not be broken with a 10 kg. Frémont hammer which gives a maximum amount of energy of 40 kgm. per sq. cm.

[1]) Mr. Charpy has contested this conclusion. He attributes the divergencies observed to want of homogeneity in the metal.

As a correlation between the results of tensile tests, of impact-tests on notched bars, and of torsion-tests, we may cite the following data given in January 1903 by the Chatillon, Commentry and Neuves-Maisons Ironworks Co, for steels containing 6% of nickel as well as for mild steels of very good quality without nickel. There were three sorts of nickel steel, numbered 1, 2 and 3. Each underwent three different treatments denoted by the

		NICKEL STEELS									MILD STEELS		
A, Heated to a dull red and cooled in the air.		No. 1			No. 2			No. 3					
B, Heated to a cherry red and cooled in the air.													
C, Quenched in water without annealing after heated to a cherry red.		A	B	C	A	B	C	A	B	C	A	B	C
With Tension	Elastic limit	70	110	125	60	70	110	40	40	70	25	25	42
	Resistance per sq. mm.	80	125	140	70	82	125	50	50	80	40	42	60
	Elongation per cent . .	20	11	10	25	18	10	30	30	13	30	30	17
	Contraction	65	53	50	70	58	55	74	75	67	65	58	58
With blows on bars nicked half-way through	Energy of rupture per sq. cm in kilogram-meters	30	19	17	40	30	18	55	55	38	22	15	35
With torsion	Moment at the elastic limit	100	160		80	100		50	54		40	40	
	Maximum moment in kilogram-meters. . .	125	165		110	135		90	95		80	85	
	Number of revolution before rupture . . .	0.8	0.4		1.15	0.6		1.55	1.1		0.7	0.7	

These approximate results are given for information and without guarantee.

letters *A*, *B* and *C*. The bars tested by tension were 13·8 mm in diameter with a gauge length of 100 mm. The notch in the bars tested by impact was circular in section according to Charpy's method.

In the paper mentioned above (Proceedings of the Institution of Mechanical Engineers, Nov. 1904, p. 1135). Messrs. A. E. Seaton and Jude have given an account of some impact-tests which they have carried out on commercial steels. They used hammer of 6 lbs (2·75 kg) weight, with a drop of 24 inches (61 cm). The test-bar was 10 cm long and of square section (12·7 mm), and rested on two supports 76 mm apart; it had one sharp notch on the one face only. It was not intended in these tests to obtain rupture at the first blow of the hammer, but to continue the blows until rupture took place, each time turning the bar back. Messrs. Seaton and Jude speak highly of this method, at least for ordinary commercial mild steels. They consider that the test approaches the actual service conditions of a large number of machine parts, which are submitted to repeated shocks. The number of blows required for rupture is on an average for ordinary mild steels (Amiranti type) 20, and may be as high as 30.

Test pieces cut out from bars broken in service, fractured after a much less number of blows.

By turning the test-piece back after each blow, it will never be bent to an appreciable degree.

Messrs. Seaton and Jude noticed in their tests that many of the steels which are in general use gave middling results in the impact-tests; if the cases in which these steels fracture in service are not numerous, the authors think that it must be due to the too large dimensions generally obtained by calculation.

Conclusions. For impact-tests on notched bars, several simple and practical pieces of apparatus of sufficient accuracy are used. These give comparative results and may be used indifferently.

The numerous tests which have been carried out do not appear to indicate a marked superiority of any of the various methods which have been used in testing notched bars by impact. Hence no one of them can be recommended to the exclusion of the others. Bars with angular notches do not give the same

results as those with rounded notches, but great precision in the shape of the notch does not appear to be of great importance; so that notches made with a saw, and without any minute precautions, will answer in many cases.

Far from finding that one special process is adopted to the exclusion of others, we find rather a tendency to consider new elements for impact tests on notched bars, such as the angle of bending after fracture, and the deformation of the broken cross section. We are thus beginning to use new experimental methods such as testing notched bars by a longitudinal blow.

The minimum numerical values of the energy absorbed in rupture, in kgm. per sq. cm, which are required for each quality of steel, has not yet been perfectly determined in practice, and these values only figure exceptionnally in the specifications.

It would seem however, that impact-tests of nicked bars have assumed a certain importance as shop-tests of metal in course of manufacture and have thus contributed to the improvement of the finished materials.

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INTERNATIONAL ASSOCIATION FOR TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 27.

THE BRINELL HARDNESS-TEST AND ITS PRACTICAL APPLICATION.

BY **J. A. BRINELL**, ENGINEER-IN-CHIEF TO THE JERNKONTOR, AND **GUNNAR DILLNER**, DIRECTOR OF THE ROYAL TESTING-STATION STOCKHOLM.
(TRANSLATED BY CH. CAPITO, LONDON.)

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The Brinell hardness-test and its practical application.

By J. A. **Brinell,** Engineer-in-chief to the Jernkontor, and Gunnar **Dillner,** Director of the Royal Testing-station, Stockholm.

(Translated by Ch. Capito, London.)

The method invented by Brinell for determining the hardness of materials has, since the time it was first laid before the International Association, come into practical use. Numerous researches have been made with the object of testing the practicability of the method, and several kinds of apparatus have been designed for carrying out the tests.

The present paper contains a summary of the results which have come under the notice of the authors. The paper is subdivided under the following headings, viz:

1. Researches concerning methods for carrying out ball-tests.
2. Researches concerning the relation between the results obtained by ball-tests and those obtained by tensile-tests.
8. Machines and measuring instruments for ball-tests.

I.

Researches concerning methods for carrying out ball-tests.

In his original tests, Brinell[1] used a hardened steel-ball which was forced by pressure into the material to be tested; the diameter of the impression was then measured, and the area of

[1] Communications au Congres International des Méthodes d'essai à Paris 1900. Tome III, p. 83.

the spherical cavity was calculated in square mm and divided into the pressure expressed in kilograms. The ratio of the pressure to the area is called the hardness-number.

In his tests, especially with iron and steel, Brinell applied a pressure of 3000 kg and used a hardened steel-ball of 10 mm diameter. Brinell remarks, in his original memoir, that for the same material, a smaller ball and the same pressure, or a higher pressure and the same ball, both give a higher hardness-number.

Trials for determining the influence due to varying the pressure and the diameter of the ball.

Benedicks[1] has closely investigated the relation between the experimental conditions with ball-testing and the resultant hardness-number. He has, amongst other things, raised the following questions:

a) In what way is the hardness-number related to the diameter of the ball used in the test?

b) In what way is the hardness-number related to the intensity of the pressure applied on the ball?

Benedicks has found the following answers to these questions:

a) A constant hardness-number for a certain metal will be obtained by multiplying the hardness-number defined by Brinell by the 5^{th} root of the radius of the ball.

The following results obtained by Brinell and Benedicks are a proof of the degree of accuracy, which this empirical rule affords.

b) From results obtained by tests with soft iron at very small loads (1—30 kg), Benedicks has drawn the curve representing the relation of the hardness-number to the load. He has then compared this curve with the corresponding curve for lead and shown, that the two curves are related to one another in the following manner, viz: that the ordinates (*H*) of the two curves corresponding to the same abscissa (*P*) bear a constant ratio to each other. From this

[1] Recherches physiques et physico-chimiques sur l'acier au carbone, Upsala 1904.

law it follows, that perfectly comparable hardness-numbers may be obtained by determining them all at the same pressure as originally suggested by Brinell.

Table Ia.

Trials carried out by Brinell.

Pressure = 2000 kg; H = hardness-number; ρ = radius of ball.

Radius of ball ρ mm	Steel No. 1		Steel No. 5		Steel No. 12	
	H	$H\sqrt[5]{\frac{}{\rho}}$	H	$H\sqrt[5]{\frac{}{\rho}}$	H	$H\sqrt[5]{\frac{}{\rho}}$
3·75	107	139	208	271	315	410
5·00	99	136	201	277	306	422
7·50	95	142	178	266	272	407

Table Ib.

Trials carried out by Benedicks.

Pressure = 500 kg; H and ρ denoting the same as in table Ia. Specimen: Electro-steel from Gysinge; C = 1·40 per cent.

ρ	H	$H\sqrt[5]{\frac{}{\rho}}$
1·60	306	366
2·49	288	346
2·99	277	344
3·97	263	346
5·00	253	350
6·37	239	346

The fact that the hardness-curves of unlike metals are affine, makes it possible within certain limits to deduce from a hardness-number, determined at a certain pressure, the value of the hardness-number at a certain other pressure. This is due to the following property of affine curves, viz: that for the same abscissa (P), fig. 1.

the tangents to the points of the different affine curves all intersect at the same point on the axis of abscissae and at the same distance, l, from the origin. The curves may therefore be substituted by their respective tangents within certain limits, which depend on the desired accuracy. It thus follows that the hardness-number, H, which has been determined at a certain pressure, P, can be reduced to the normal value, H_0, corresponding to the pressure, P_0, by means of the following formula:

$$H_0 = H \times \frac{l + P_0}{l + P}$$

P and P_0 must however lie between the limits P_1 and P_2. The value of l which depends on the mean-value, $\frac{P_1 + P_2}{2}$, has been determined by Benedicks, table II, from experiments carried out by himself and others.

Table II.

Values of limits		$\frac{P_1 + P_2}{2}$	l
P_1	P_2	approximate	
140	500	300	3700
500	4000	2000	17000

Impact ball-tests.

Brinell has pointed out, in his original paper, that in the case of larger specimens, it might be impossible to place them in the testing machine, if static pressures, gradually applied to the ball, are to be used. In such cases, however, there is as a rule no difficulty in making impressions of the ball in the specimen by applying dynamic pressures (impact). Brinell has carried out a series of experiments for the purpose of determining the relation between impact ball-tests and the ordinary static ball-tests.

The experiments were carried out in the following manner; firstly, an ordinary ball-test (static load = 3000 kg) was made,

and the diameter of the impression was measured; secondly, by repeated trials, the height through which a weight of 5 kg must fall so as to make a similar impression with a 10 mm ball, was determined. This height was found to be 450 mm. Tests made with iron, containing 0·10 per cent of carbon, showed that energy of impact, amounting to about 2·25 kilogram-metres, corresponds to a static load of 3000 kg gradually applied. One might have expected that this relation would also hold good for other per-

Table III.

Percentage of carbon	Impact ball-test (energy of impact = 2·25 kilogram-metres)			Ball-test with a gradually applied load			Difference	
	Diameter of impression	Area of spherical impression	Hardness-number	Diameter of impression	Area of spherical impression	Hardness-number	$H-H_s$	$\frac{H-H_s}{H}$
	mm	mm²	H_s	mm	mm²	H		
0·10	5·50	25·8931	116	5·50	25·8931	116	—	—
0·20	5·40	24·8720	121	5·15	22·4357	134	13	0·103
0·30	5·20	22·9085	131	4·75	18·8527	159	28	0·176
0·40	5·10	21·9629	137	4·70	18·4286	163	26	0·159
0·50	5·00	21·0455	143	4·50	16·8044	179	36	0·201
0·60	4·85	19·7185	153	4·15	14·1749	212	59	0·278
0·70	4·65	18·0186	166	4·10	13·8262	217	51	0·235
0·80	4·50	16·8044	179	3·90	12·4407	241	62	0·257
0·90	4·25	14·8943	202	3·45	9·6478	311	109	0·350
1·00	4·15	14·1749	212	3·45	9·6478	311	99	0·318

centages of carbon, but experiments show that this is not the case. On the contrary, it appears that the harder the metal is, the greater is the difference between the hardness-number determined by using a load gradually applied and that determined by impact. The difference is this, that the hardness-numbers found by impact tests are higher than those found by static ball-tests. The results of the trials are shown in table III.

The hardness-number, H_s, in table III, has been calculated by assuming that the effect of the ball on the test-piece, due to the energy of impact, is equivalent to that of a gradually increasing static load of 3000 kg. This method has been adopted for the purpose of pointing out the difference in the results obtained by the two methods of testing, which the diameters of impression alone do not clearly show.

The fact that the hardness-number obtained by impact ball-test is not the same as that obtained by static ball-test, seems to be a definite obstacle to applying the former method. This obstacle has, however, been overcome by Mr. J. O. Roos, Engineer at the Testing-Station connected with the Royal Technical College in Stockholm, as shown in his report on impact ball-tests. This report, which has not been published before, is supported by numerous experiments carried out by himself during the present year.

Roos's Researches.

In the calculations shown below, the following suppositions with regard to the impression of the ball in the material have been made, viz:

The load acts in the direction of the normal to the surface of the material.

The shape of the ball remains a sphere during the whole of the impression.

The reaction on each element of the surface of deformation is normal, and is taken as being constant per unit area throughout the entire surface, and equal to p kilograms.

The radius of the ball $= \rho$.

The radius of impression $= r$.

The area of the spherical surface of impression $= A$.

The depth of the impression $= h$.

In the case of a static load of P kg we have

$$P = p \pi r^2 \quad \ldots \ldots \ldots \quad 1)$$

The energy of impact applied on the ball $= Q$.

The effective energy of impact $= k Q$, where k is an unknown and possibly a variable coefficient, which depends on the installation, dimensions etc. of the testing-machine.

The ball will be in equilibrium when the effective energy of impact equals the energy of deformation.

At any moment during impact, the radius of impression equals r_1 and therefore the total reaction equals $p \pi r_1^2$. To an increment of depth, dh, corresponds an increment of energy of deformation $= p \pi r_1^2 \, dh$; hence the total energy of deformation $= \int_0^h p \pi r_1^2 \, dh = p$ times the spherical volume of impression.

$\therefore k Q = p$ times the spherical volume of impression.

$$\therefore k Q = p \pi h^2 \left[\rho - \frac{h}{3} \right]$$

But $A = 2 \pi \rho h$, hence

$$k Q = \frac{p A^2}{4 \pi \rho} \left[1 - \frac{h}{3 \rho} \right]$$

$$\therefore \frac{A^2}{Q} = \frac{k 4 \pi \rho}{p} \left[1 + \frac{h}{3 \rho} \right] \quad \ldots \ldots \quad 2)$$

For the same testing-machine and the same ball-diameter, the right hand side of equation (2) may be considered constant, as the term $\frac{h}{3 \rho}$ is very small with large impressions and may therefore be neglected.

Impact ball-tests, with a weight of 5 kg falling through different heights, have been carried out on two materials containing respectively 1·20 and 0·10 per cent of carbon. The results are shown in table IV.

Table IV.

Height of fall cm	1·20 per cent of carbon		0·10 per cent of carbon	
	Diameter of impression mm (two tests)	$\frac{A^2}{Q}$	Diameter of impression mm (two tests)	$\frac{A^2}{Q}$
5	2·625	1·21	3·20	2·73
10	3·10	1·20	3·80	2·76
15	3·45	1·24	4·20	2·81
20	3·65	1·18	4·525	2·89
30	4·05	1·21	5·00	2·95
40	4·375	1·25	5·35	2·97
50	4·575	1·21	5·65	3·01

If we consider that an error of one percent in measuring the diameter of the impression will result in an error of 4 per cent in $\frac{A^2}{Q}$, we may with sufficient accuracy say, that for the same material and the same diameter of the ball, the relation between the spherical area of the impression of the ball and the applied energy of impact, may be expressed by the formula

$$\frac{A^2}{Q} = \text{a constant} \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad 3)$$

In the following calculations for determining the relation between the impression of the ball by a static load and that by impact, suffix S is used to denote impact.

In the case of a static load, we have

$$P = p\pi r^2$$

but $A = 2\pi\rho h$ and $r^2 = h\,(2\rho - h)$, hence

$$\pi r^2 = A\left(1 - \frac{h}{2\rho}\right), \text{ and } P = pA\left(1 - \frac{h}{2\rho}\right)$$

In the case of impact, we have found

$$\frac{A_s^2}{Q} = \frac{k 4\pi\rho}{p}\left(1 + \frac{h}{3\rho}\right) \quad \ldots \ldots \ldots \ldots \quad 2)$$

By eliminating p between the two last equations, we obtain

$$\frac{A_s^2}{A} = \frac{kQ}{P} \times 4\pi\sigma\left(1 - \frac{h}{6\rho}\right)$$

Now the definitions of the hardness-numbers H and H_s are

$$H = \frac{P}{A} \text{ and } H_s = \frac{P_s}{A_s}$$

Hence we have

$$\frac{H_s^2}{H} = \frac{P_s^2}{kQ} \times \frac{1 + \frac{h}{6\rho}}{4\pi\rho}$$

The right hand side of this expression does not contain any quantity, which depends on the material to be tested; if therefore the energy of impact Q and the pressure P_s are the same in the different cases, we may expect to find that

$$\frac{H_s^2}{H} = \text{a constant} \quad \ldots \ldots \ldots \ldots \quad 4)$$

In order to find out whether the theoretical relation between the hardness-number (H_s), obtained by impact, and that (H) obtained by a static load is in agreement with actual tests, 27 impact-tests with steels from three Swedish iron-works, marked H, K and S, have been carried out. The results of these tests are shown in table V.

The impact-tests, table V, were carried out with a weight of 5 kg falling through a height of 450 mm which, according to Brinell, produces a spherical impression equal to that due to a static pressure of 3000 kg. The hardness-number for impact has been calculated in the same manner as mentioned above with Brinell's impact-tests. All the specimens were annealed at 860° before tested.

Table V.

Mark of Mill	Percentage of carbon contained in the steel	Hardness-number		
		for impact H_s	for static load H	$K = \frac{H_s^2}{H}$
S	0·1	109	107	111
K	0·1	109	107	111
S	0·2	114	118	110
K	0·2	114	116	112
S	0·3	127	137	117
K	0·3	126	134	118
H	0·3	116	114	118
S	0·4	128	140	117
K	0·4	124	131	117
H	0·4	126	134	118
S	0·5	134	153	117
K	0·5	131	146	118
H	0·5	131	146	118
S	0·6	140	170	115
K	0·6	141	170	118
H	0·6	131	149	115
S	0·7	158	187	125
K	0·7	146	179	119
H	0·7	146	174	122
S	0·8	166	205	134
K	0·8	163	202	131
H	0·8	156	187	130
S	0·9	163	200	133
K	0·9	165	223	122
H	0·9	163	198	134
S	1·0	169	228	125
H	1·0	163	196	135

The experimental data show that the ratio $\frac{H_s^2}{H}$ which ought to be constant according to Roos' theory, increases with the

percentage of carbon. For 0·1 and 0·2 per cent of carbon, the ratio is 110—112; for 0·3 to 0·6 per cent of carbon, the ratio is 115—118; and finally, when the percentage of carbon is greater than 0·6, the ratio $\frac{H_s^2}{H}$ varies irregularly between the values of 113 and 135. It is thus possible to determine by calculation the static hardness-number from that obtained by impact-test with an accuracy as mentioned, provided the percentage of carbon does not exceed 0·6.

Impact ball-tests as well as static ball-tests have been carried out with the object of investigating the influence of annealing on the relation between the two hardness-numbers. These tests were made with two steels from some other Swedish iron-works and containing 0·16 and 0·79 per cent of carbon respectively. The results are shown in table VI.

Table VI.

Temperature of annealing	Steel Nr. 1 (0·16 per cent of carbon)			Steel Nr. 2 (0·79 per cent of carbon)		
	H_s	H	$\frac{H_s^2}{H}$	H_s	H	$\frac{H_s^2}{H}$
350°	125	140	112	183	274	122
550°	126	143	111	189	277	129
750°	121	129	113	177	252	124
850°	125	140	112	179	241	133
1000°	121	131	112	177	252	124
1200°	123	133	112	183	266	126

The above experimental data show that for the same material the ratio $\frac{H_s^2}{H}$ is constant and independent of the temperature of annealing.

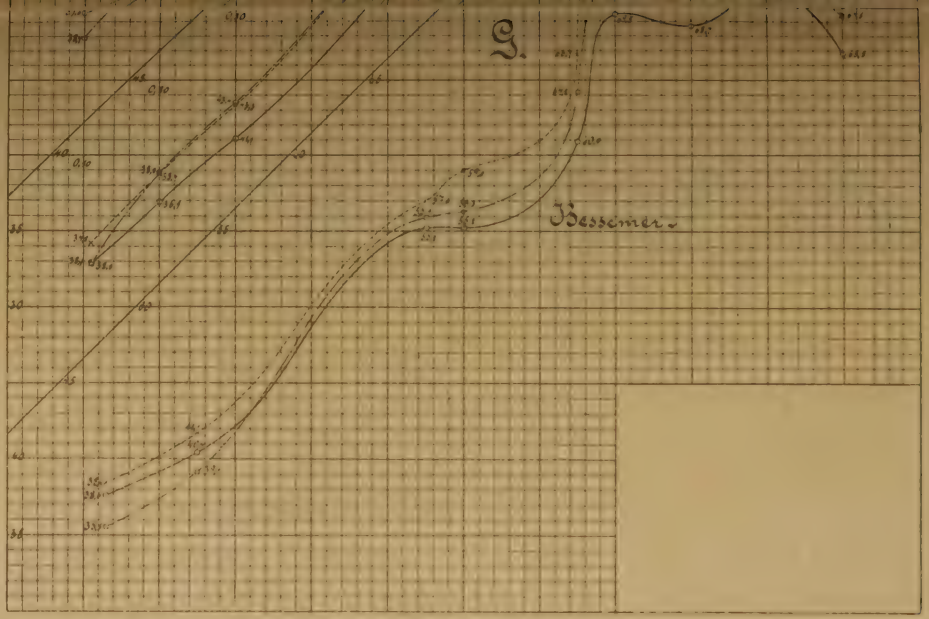
II.

Researches concerning the relation between the results obtained by ball-tests and those obtained by tensile-tests.

Brinell points out in his original paper that there exists, especially in the case of steels poorer in carbon than 0·8 per cent, a certain relation between the hardness-number of the material and its tensile strength. Brinell has found that for warm rolled steel from Fagersta, the ultimate tensile strength of the material may be obtained by multiplying the hardness-number by 0·346.

As this observation is of the greatest interest so far as it gives a cheap and ready means for determining the ultimate tensile strength, without having to shape the test-piece beforehand, it is only natural that the said relation between the hardness-number and the tensile strength should be the object of a thorough research.

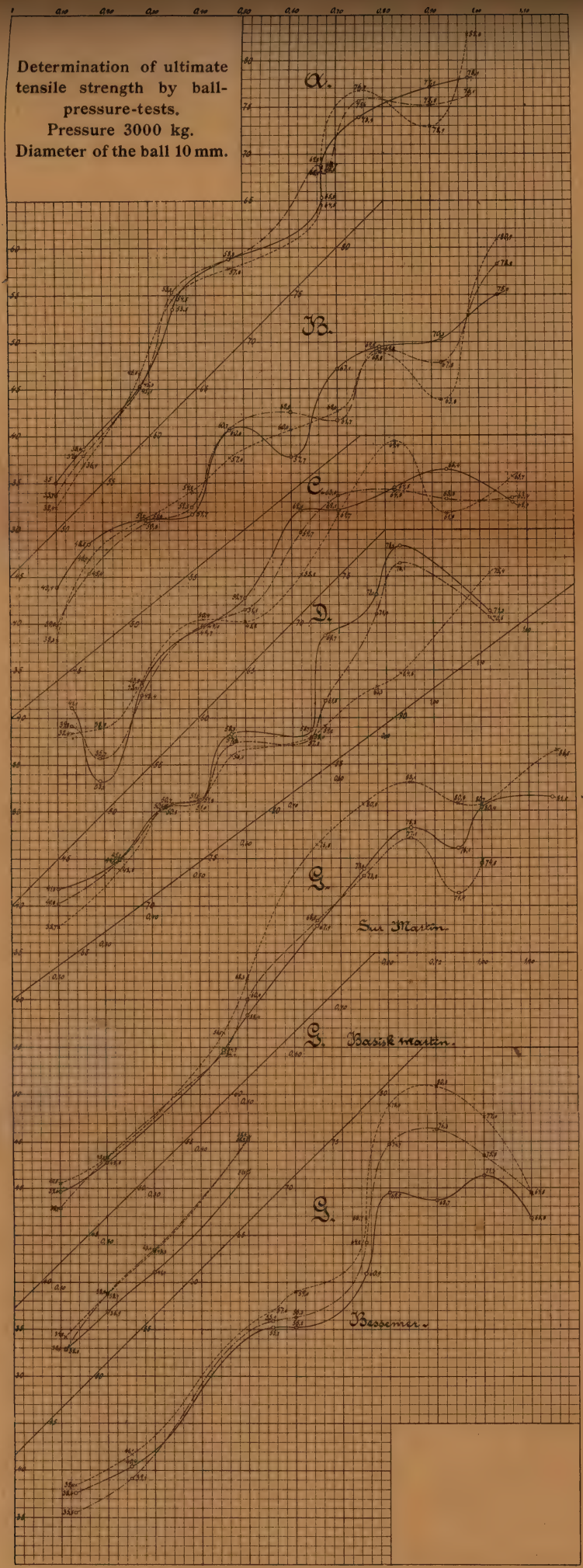
A series of tests with this object has been carried out by Mr. Dillner of the Testing station connected with the Technical High-School in Stockholm; the expenses have been borne by the Jernkontor. The object of the trials was to find out how far the number 0·346, as found by Brinell for Fagersta-steel, holds good for all Swedish steels. Trials were carried out altogether with 7 series of steels containing from 0·10 to 1·20 per cent of carbon and taken from 5 different kinds of specimens manufactured by the acid Bessemer process, as well as by the acid and basic Martin process; all the specimens were rolled warm and afterwards annealed at 860° C. Hardness-tests and tensile-tests were made with each steel, and the ratio of the ultimate tensile strength to the hardness-number, the so called reduction coefficient, was calculated. The experimental data show, that this coefficient diminishes with the percentage of carbon contained in the specimen, but this diminution was as near as possible the same with steels from the different iron-works. If the same average coefficient were taken for steels with all percentages of carbon, the result would be that in the case of very soft and very hard steels, the calculated values of the ultimate tensile strength would differ too much from the actual values. One has tried to avoid this error by determining an average coefficient for carbon percentages lower



———————— Ultimate tensile strength found by ball-tests, pressure parallel with the fibres.

– – – – – – – – – – – – „ „ „ „ „ „ „ across the fibres.

—·— — ·— — ·— „ „ „ „ „ tension-tests.



——————— Ultimate tensile strength found by ball-tests, pressure parallel with the fibres.

– – – – – – – – – – – – „ „ „ „ „ „ „ „ „ across the fibres.

—·—·—·—·—·— „ „ „ „ „ „ tension-tests.

than 0·5, and another and smaller coefficient for carbon percentages above 0·5. This method would, however, require a previous knowledge of the amount of carbon contained in the material, which is to be subjected to ball-tests. As a rule, this knowledge is not at hand, and it is therefore better to take a certain hardness-number as the limit for determining which of the coefficients is to be used in each case. The hardness-number 175 has been chosen as this limit, and two mean-coefficients have been calculated, one to be used when the hardness-number is less than 175 and the other for hardness-numbers equal to or greater than 175.

It was found during the trials, that very different hardness-numbers were obtained according to whether the direction of the pressure on the ball was parallel with the fibres or across the fibres. In the former case, we obtain greater hardness-numbers and therefore smaller coefficients than in the latter case. In all Brinell's trials, the direction of the pressure on the ball was parallel with the fibres. In the practical application of the ball-test, i. e. testing-materials for structures, it is only in exceptional cases that the pressure on the ball is to be applied in the direction of the fibres; on the contrary, in most cases the tests are carried out on the side of the plate, or of the beam, etc. For this reason, the hardness tests were carried out in both directions, and the following coefficients were found for Swedish iron and steel.

Hardness-number.	Coefficients	
	Ball-pressure across the fibres.	Ball-pressure parallel with the fibres.
Below 175	0·362	0.354
Above 175	0·344	0·324

Diagram I shows the difference between the values of the ultimate tensile strength found by direct tests and those found by calculation with the coefficients given above. These curves show that in the case of softer material (containing less than 0·5 per cent of carbon) the two sets of values agree very well; the mean deviation being 3·3 per cent. For materials containing higher

percentage of carbon than 0·5 the mean deviation is higher (6·1 per cent). The greater deviation, which exists with higher percentages of carbon, is however of little significance, as this method is chiefly important for testing-materials containing lower percentages of carbon.

Mr. Dillner has also checked the values obtained by applying these coefficients to rolled Swedish section-iron and without taking any particular trouble about the annealing of the material. For this purpose, he has carried out 30 tests with plate-iron and section-iron supplied by two iron-works, which had not delivered the material used for determining the coefficients.

The experimental data given in table VII show that the ball-test may be used to determine the breaking load even for materials used in building-structures, and which are not annealed; and also that the mean-coefficients, determined by means of annealed steel, may be used for similar iron. The mean deviation between the values of the ultimate tensile strength as found by direct tests, and those found by means of ball-tests, amounts to 4·4 per cent in the case of the specimens from mills E and F. The results in this case agree therefore as well as the results in the case of annealed specimens.

Trials have also been carried out with the object of investigating whether the same mean-coefficients could be used to calculate the ultimate tensile strength of tempered and cold-rolled steel specimens. Generally, the results have innot been satisfactory. The trials have, however, shown that with steel treated in this manner there exists a certain relation between hardness and ultimate tensile strength; and it even seems likely that by applying other coefficients the hardness-number may also be used in this case to determine the ultimate tensile strength of the materials.

Trials carried out by Mr. Charpy.

The trials, referred to above have all been made with Swedish steel. It is, therefore, of great interest to study the results of similar trials with steels from other countries. In this respect, good information is offered by the results of trials, which Mr. Charpy presented to the meeting of French Members of the International

Table VII.

Hardness-number across fibres	Ultimate tensile strength kg per mm²	Ultimate tensile strength calculated from hardness-number kg per mm²	Mean coefficient used	Remarks
107	37.1	38.7	0.362	
105	36.8	38.0	,,	Boiler-plate
109	38.1	39.5	,,	
107	38.0	38.7	,,	Ships-plate
131	44.1	47.4	,,	
137	44.9	49.6	,,	Boiler-plate
117	41.1	42.4	,,	
118	41.9	42.7	,,	Ships-plate
134	43.2	48.5	,,	
134	44.6	48.5	,,	Boiler-plate
131	46.4	47.4	,,	
136	46.1	49.2	,,	Ships-plate
148	51.3	53.9	,,	Frame-iron
146	50.3	52.9	,,	60×60×6 mm
113	37.5	40.9	,,	Angle-iron
106	37.5	38.4	,,	75×75×10 mm
149	52.3	53.9	,,	Angle-iron
153	52.4	55.4	,,	55×55×6 mm
159	55.2	57.6	,,	
163	55.4	59.0	,,	Rail
116	39.5	42.0	,,	
114	39.0	41.3	,,	Channel-iron
116	40.0	42.0	,,	
116	40.2	42.0	,,	Frame-iron
92	32.7	33.3	,,	6 mm
94	33.2	34.0	,,	6 ,,
105	35.6	38.0	,,	7 ,,
99	36.4	35.8	,,	7 ,,
118	42.2	42.7	,,	8 ,,
116	41.1	42.0	,,	8 ,,
109	39.0	39.5	,,	Angle-iron 9 ,,
107	39.7	38.7	,,	9 ,,
103	38.8	37.3	,,	12 ,,
105	39.0	38.0	,,	12 ,,
95	36.6	34.4	,,	15 ,,
103	39.5	37.3	,,	15 ,,

Hardness-number across fibres	Ultimate tensile strength kg per mm²	Ultimate tensile strength calculated from hardness-number kg per mm²	Mean coefficient used	Remarks	
116	44.4	42.0	0.362	Angle-iron	10 mm
116	44.2	42.0	,,		10 ,,
105	37.4	38 0	,,	Flat bar-iron	11 ,,
105	37.7	38.0	,,		11 ,,
114	39.3	41.3	,,	Boiler-plate	8 ,,
112	37.3	40.5	,,		8 ,,
109	39.1	39.5	,,		10 ,,
109	41.5	39.5	,,		10 ,,
112	40.5	40.5	,,		12 ,,
112	39.1	40.5	,,		12 ,,
105	37.4	38.0	,,		14 ,,
105	37.7	38.0	,,		14 ,,
107	40.1	38.7	,,		16 ,,
112	40.3	40 5	,,		16 ,,
114	40.0	41.3	,,	Ships-plate	8 ,,
107	40.1	38.7	,,		8 ,,
109	38.5	39.5	,,		10 ,,
118	41.1	41.7	,,		10 ,,
105	37.8	38.0	,,		12 ,,
101	35.8	36 6	,,		12 ,,
105	35.5	38.0	,,		14 ,,
95	36.3	34.4	,,		14 ,,
107	38.3	38.7	,,		16 ,,
109	40.7	39.5	,,		16 ,,

Association for Testing Materials on July 21st 1902. These trials were made with French steels, some of which contained chromium and nickel, and others were tempered. The tensile tests were made with cylindrical test-pieces, diameter 13·8 mm; after rupture had taken place, the enlarged end of the test-piece was cut through and the ball-test was carried out on the polished section. The diameter of the ball was 8 mm, and the pressure was 4000 kg.

Table VIII.

A. Steel with hardness-number smaller than 175.
(According to Charpy)

Ultimate tensile strength kg per mm² O	Hardness-number H	$\frac{O}{H} = K$	Ultimate tensile strength calculated from hardness-number kg per mm²	Error per cent
30·4	80·0	0·380	28·1	7·6
32·1	91·2	0·395	32·0	0·3
35·4	95·6	0·370	33·6	5·1
36·4	92·0	0·395	32·3	11·3
36·8	100·8	0·365	35·4	3·8
36·8	106·4	0·345	37·3	1·4
38·1	112·4	0·339	39·5	3·7
38·8	110·0	0·352	38·6	0·5
39·1	114·4	0·342	40·2	2·8
40·1	118·0	0·340	41·4	3·2
41·1	114·8	0·358	40·3	2·0
41·5	114·4	0·363	40·2	3·1
41·5	120·0	0·346	42·1	1·4
42·1	113·6	0·371	39·9	5·2
42·8	116·0	0·369	40·7	4·9
41·3	126·4	0·341	44·4	3·0
43·8	130·0	0·337	45·6	4·1
44·1	121·0	0·364	42·5	3·6
44·1	130·0	0·339	45·6	3·4
46·8	132·4	0·353	46·5	0·6

Ultimate tensile strength kg per mm² O	Hardness-number H	$\frac{O}{H} = K$	Ultimate tensile strength calculated from hardness-number kg per mm²	Error per cent
46·8	137·2	0·341	48·2	3·0
46·8	141·4	0·331	49·6	6·0
49·1	140·8	0·346	49·4	6·1
49·1	142·0	0·345	49·8	1·4
50·1	146·8	0·341	51·5	2·8
50·8	152·4	0·333	53·5	5·3
51·1	148·6	0·344	52·1	2·0
51·8	146·8	0·353	51·5	0·6
52·1	146·8	0·355	51 5	1·1
54·2	162·9	0·333	57·2	5·5
54·8	153·1	0·356	53·7	2·0
54·8	155·2	0·353	54·5	0·5
55·1	158·7	0·347	55·7	1·1
55·8	165·75	0·337	58·2	4·3
56·4	162·9	0·346	57·2	1·4
56·4	166·5	0·339	58·4	3·5
56·8	166·5	0·341	58·4	2·8
58·1	171·75	0·338	60·3	3·8
59·4	169·5	0·350	59·4	0·0
60·8	174·2	0·349	61·1	0·5
		0·351		3·1

Table IX.

B. Steel with hardness-number greater than 175 (according to Charpy).

Ultimate tensile strength kg per mm² O	Hardness-number H	$\frac{O}{H} = K$	Ultimate tensile strength calculated from hardness-number kg per mm²	Error per cent
57·1	175·0	0·326	58·8	3·0
59·4	175·15	0·339	58·9	0·9
61·5	175·9	0·350	59·1	2·3
62·1	188·65	0·329	63·4	2·1
62·7	182·7	0·343	61·4	2·1
64·4	185·25	0·347	62·3	3·3
65·4	187·8	0·348	63·1	3·5
66·1	196·3	0·337	66·0	0·2
66·8	200·0	0·334	67·2	0·6
68·1	204·0	0·334	68·5	0·6
69·4	199·0	0·348	66·9	3·6
69·4	209·0	0·332	70·2	1·1
70·4	201·0	0·350	67·5	4·1
78·5	244·5	0·321	82·1	4·5
97·5	291·5	0·334	97·9	0·4
102·0	308·0	0·331	103·5	1·5
109·5	338·0	0·324	113·6	4·0
121·6	362.0	0·336	121·6	0·0
121·6	366·0	0·332	123·0	1·2
		0·336		2·1

The direction of the pressure was with the fibres. The results of a total of 60 tests are shown in Table VIII.

Two mean-coefficients have also been determined from Charpy's results, one for steels with a hardness-number smaller than 175, and another for steels with a hardness-number greater than 175. The two mean-coefficients worked out to be 0·351 and 0·336 respectively; they agree fairly well with those found for Swedish steel. By means of these mean-coefficients and the hardness-numbers found by Charpy, values of the ultimate tensile strength have been worked out. These values, together with their deviations (in per cent) from the actual values of the ultimate tensile strength, are shown in table VIII.

The results given in Tables VIII and IX show that the mean-coefficients deduced from Charpy's trials do not differ sensibly from those found by testing Swedish steel. The values of the ultimate tensile strength, calculated from Charpy's tests vary almost to the same extent as those found by Dillner's trials.

Ball-testing as a means of determining the ultimate extension and ductility of steel.

In his original paper, Brinell hints at the possibility, of determining by particular methods, — besides the ultimate tensile strength — also the ultimate extension and ductility of steel. He mentions, however, that up to the time of writing his paper he does not possess sufficient experimental data to be able to say with certainty how far accurate results could be obtained by such determinations.

Dillner has since then closely investigated this question and carried out some extensive trials in the manner indicated by Brinell. The results show that the values of ductility and of ultimate extension deduced from ball-testing, differ from their actual values, determined by direct tests, to the amount of 10—20 per cent.

Similar results have been obtained by Breuil, according to a paper presented by him to the meeting of the French Members of the International Association for Testing Materials on the 21st of July 1902.

III. Machines and measuring instruments for ball-tests.

Any testing-machine may be used for carrying out ball-tests, provided that it can be accurately adjusted to a certain desired load.

Since this method of testing has come into general practical use, a large number of special machines have been designed for the purpose; it would, however, lead us too far to describe all the different designs, and the authors have therefore limited themselves to describing a few of them in some detail.

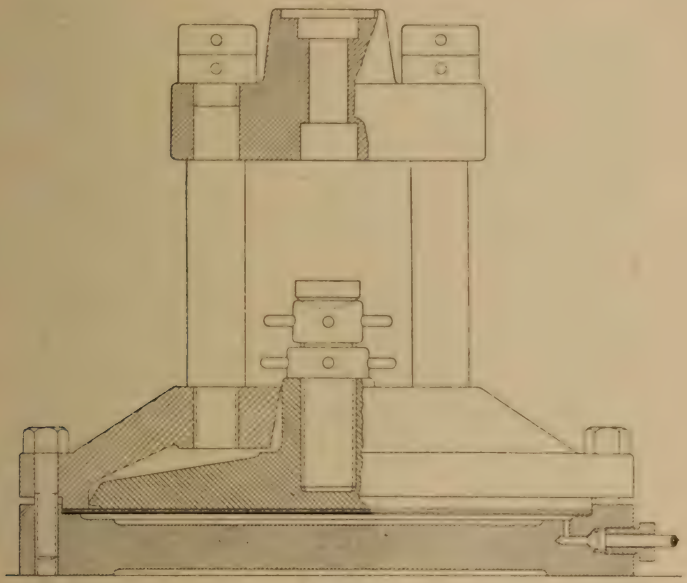


Fig. 1.

Prof. A. Martens has designed a machine, fig. 1, for this purpose for the Royal Testing-Station, Groß-Lichterfelde. A diaphragm-box is used for fixing the pressure. This box is a strong metallic vessel closed by a thin diaphragm of India-rubber or thin sheet-brass. A strong cover placed on the top of the diaphragm is pressed upwards by high pressure water contained in the diaphragm-box; the ball which is placed on the top of the cover, will thus be pressed into the material to be tested. The object of the screws is to adjust the ball against the test-piece at the

commencement of the test. The pressure applied on the ball is measured by a pressure-gauge connected with the diaphragm-box. A hollow space is left in the uppermost part of the machine for inserting an apparatus by which the depth of the impression made by the ball can be measured. The construction of this apparatus is described in the proceedings of the «Verein zur Beförderung des Gewerbefleißes», 1906, page 74.

The «Aktiebolaget Alpha» («Alpha» Company) of Stockholm has designed a good ball-testing machine, which is easy to mani-

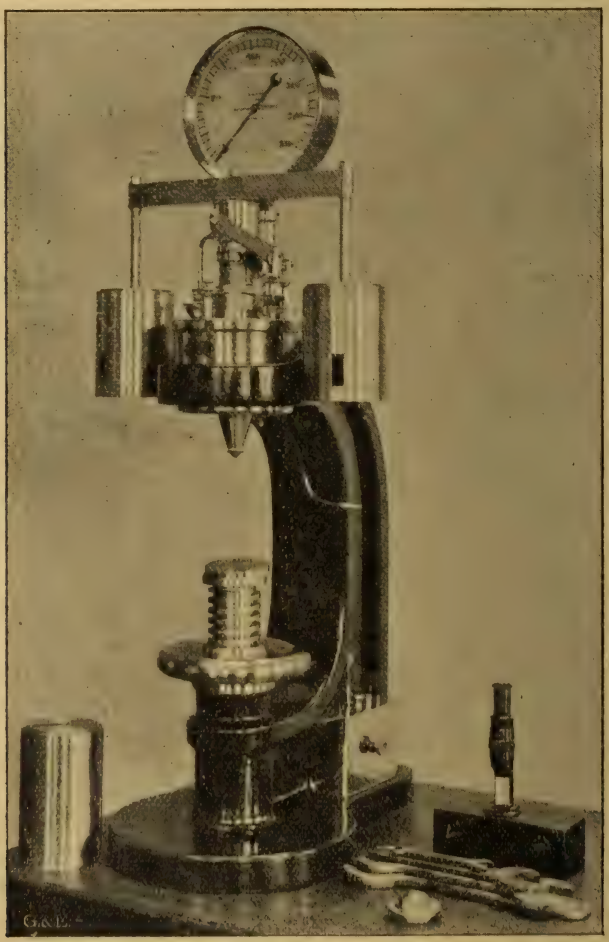


Fig. 2.

pulate (fig. 2). It is a hydraulic press, the steel-ball being fixed on the piston. The test-piece is placed on an adjustable table provided with ball-bearings. The piston fits into the cylinder without packing. The pressure in the hydraulic cylinder is produced by a small hand-pump and is measured by a pressure-gauge. The machine is also provided with an apparatus for con-

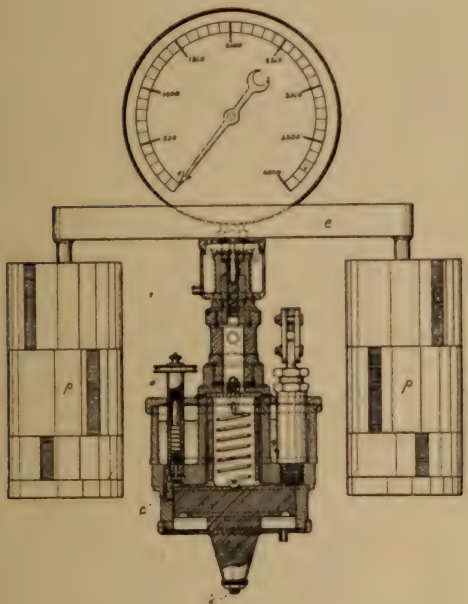


Fig. 2a.

trolling the pressure, so that the desired pressure cannot be exceeded. This latter apparatus is directly connected to the pressure cylinder and consists of a smaller cylinder inside which a piston moves frictionless. This smaller piston is loaded with dead weights corresponding to the desired pressure, and when the latter is reached, the piston together with the weights will be lifted and will remain floating.

Brinell has also designed a portable apparatus for impact ball-tests (fig. 3). The apparatus consists of a steel-tube in which a weight of 5 kg can move. The weight is held in position at the

topmost part of the tube by a hook fixed to a small transversal spindle, which ends in a button outside the tube. The apparatus is accurately placed in a vertical position. By pressing the button the weight will be released and will fall, without friction, direct upon a piston placed at the bottom of the tube; a small steel-ball is fixed on the end of the piston-rod. The height of the fall and the size of the weight are so adjusted, that the impression made by the ball in a specimen of iron, containing 0·1 per cent of carbon, is equal to the impression which would be caused by a static load of 3000 kg.

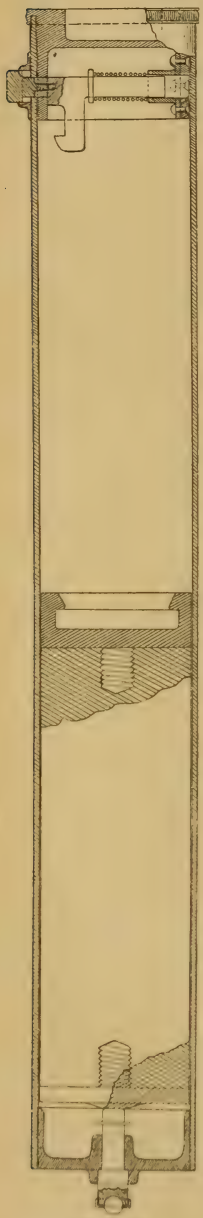


Fig. 3.

Another apparatus for impact ball-testing has been designed by Guillery (fig. 4). It

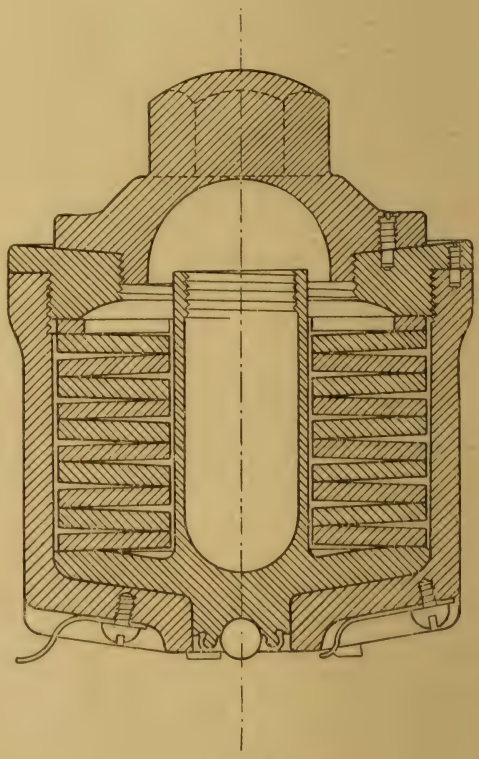


Fig. 4.

consists of a hollow piston, which can move inside a cylinder. The latter is closed at the top and the cover ends in a head, which receives the shock of the impact. The steel-ball is fixed in a special manner to the bottom of the piston. Inside the

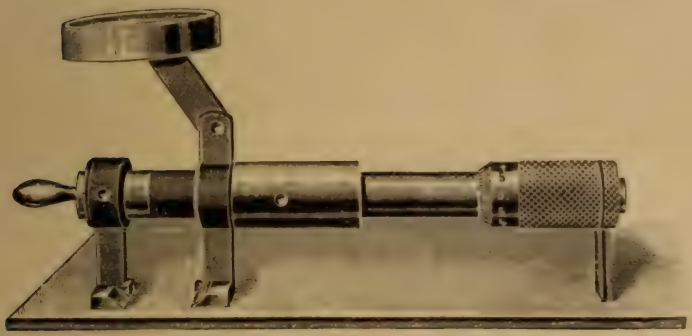


Fig. 5.

cylinder and surrounding the greater part of the piston are placed a number of Belleville-springs, one above the other, filling up the entire space. By applying a sufficiently strong force of

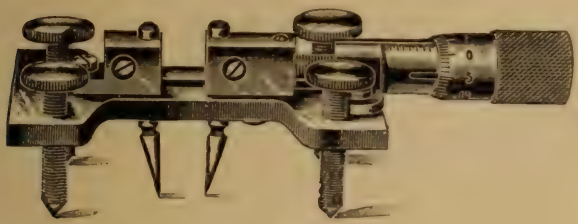


Fig. 6.

impact the springs will be so much compressed that the bottom part of the cylinder will come to rest on the test-piece, while at the same time the steel-ball will be pressed into the test-piece. It is evident that by a proper adjustment of the springs, it is also possible with this apparatus to obtain ball impressions, which are equal to those produced by a static load of 3000 kg.

Attention must be called to the importance of placing the test-piece on an inelastic foundation when submitting it to impact ball-tests.

The firm of Huber & Wagner, St. Ludwig in Alsace, have designed some instruments (fig. 5 and 6) for measuring the large impressions made by a 19 mm ball, which is used in Germany

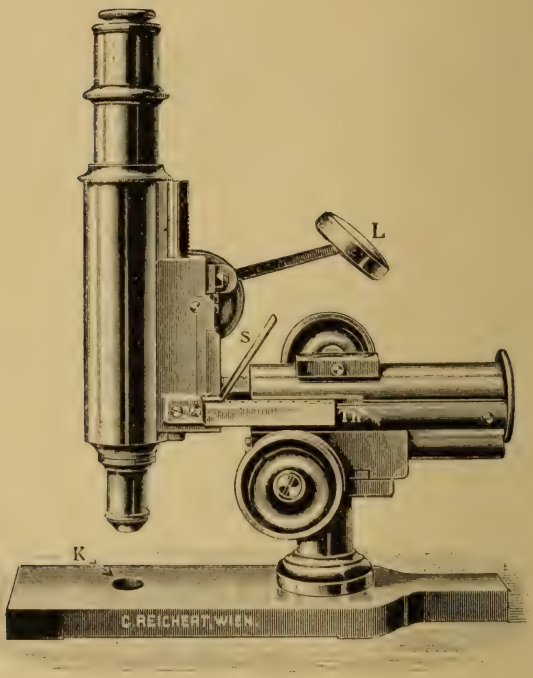


Fig. 7.

for testing the hardness of rails. This kind of apparatus does not however, suffice for measuring smaller impressions; in the latter case different kinds of microscopes are used. Brinell has designed a microscope for this purpose which has been largely used, and which is sold by the firm of C. Reichert of Vienna. As will be

seen (fig. 7), the microscope is provided with a vernier and is adjustable in two directions. The „Alpha“ Company of Stockholm has also placed a microscope on the market, which is provided with an eye-piece micrometer. The accurate position of the instrument relative to the ball-impression is adjusted by simply moving the microscope by hand, without the application of screws and the like.

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 36.

ON THE INTERNATIONAL RESEARCHES IN MACROSCOPIC EXAMINATION (ETCHING TESTS) OF IRON.

BY K. K. REGIERUNGSRAT
WILHELM AST, CHIEF-ENGINEER
OF THE KAISER FERDINANDS-NORD-
BAHN, VIENNA, AUSTRIA.
(TRANSLATED BY R. N. LUCAS.)

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On the International Researches in macroscopic examination (etching tests) of iron

by

k. k. Regierungsrat **Wilhelm Ast,**

Chief-Engineer of the Kaiser Ferdinands-Nordbahn, Vienna, Austria.

(Translated by R. N. Lucas.)

The President of the International Association for Testing Materials has honoured me with the invitation to furnish a report regarding the fourth Congress to be held in Brüssels in 1906, on „The international results obtained up to the present date in regard to the macroscopic examination (etching tests) of iron".

I have much pleasure in conforming to this proposal, more particularly as I have come to the conclusion as the result of experience that the macroscopic structure and resistance to strain of iron and the relationship in which the two stand to one another is of considerable practical importance. In order to base the observations on as wide a groundwork of experience as possible I have with the consent of the President directed a list of questions to the members of the Committee with the request that the questions might be answered as fully as possible.

The list of questions is as follows:

a) Are macroscopic examinations of iron undertaken in your country?

b) Are such examinations only made in the laboratory for scientific purposes or are they also made by manufacturers and users of iron?

c) What are the results which have been obtained by the examinations?

d) Have macroscopic examinations been included in specifications in your country?

e) Is evidence of such examinations to be found in the literature of your country?

19 answers were received to this list of questions from eight different countries which are represented on the Association for which I beg to thank the senders most sincerely.

The answers to the questions are given below literally. Two of the answers, however, have been omitted as they referred to the macroscopic examination of iron.

A. Replies to Questions.

1. Danish Testing Institute. Professor H. T. Hannover, Copenhagen.

In continuation of my letter of 2nd June 1903, I beg to inform you that after distributing the list of questions sent by Mr. Councillor Ast among the authorities in the country I represent I have received the answer that macroscopic examinations of iron have not been made by them and it does not seem probable that other people in Denmark have made them either.

It was only in this Institution that some years ago some experiments were made at the suggestion of the President of the Union with iodine etching for the purpose of trying to ascertain the causes of certain exceptional cases of corrosion. The results however, were not satisfactory.

2. Engineer Fritz Lürmann jr., Osnabrück.

a) Etching tests are employed in all works in Germany where operations are conscientiously conducted, for the purpose of controlling the work of the factories.

b) Etching tests are employed for scientific purposes, they are also used by manufacturers to control the quality of the steel and other materials which they turn out.

c) The electric conductivity of mild steel depends upon its homogeneity while the weldability of rail steel also depends upon the same quality to a certain extent.

d) I am not aware that macroscopic examinations of iron have been included in contract specifications in Germany; this examination could not be advisably included in such contracts. To be able to judge of the various qualities of material satisfactorily from etching tests requires a long period of conscientious study as well as practical experience of many years, qualifications which inspectors seldom have opportunities of acquiring.

Steel and mild steel should in my opinion only be accepted after practical testing, the tests employed being such as correspond to the purposes to which the material is to be adapted. In addition, the etching tests are carried out in a variety of ways and by a variety of different methods. The same sample of steel or mild steel can give good or bad results according to the method of etching adopted. The quality of the material is much more difficult to determine by the etching test at present than by analysis.

e) See «Stahl und Eisen«.

3. The Royal Mechanical Testing Institute, Charlottenburg, Geh. Regierungsrat Prof. A. Martens.

As the testing institution has its own metallurgical laboratory it of course carries out macroscopic examinations and etched tests on an extended scale. The results that have been obtained have to a certain extent been reproduced in the literature of the subject.

The view of the Institute is that the macroscopic examination (etched test) referred to in the list of questions is the usual long continued etching with hydrochloric acid in which the material is strongly attacked and partially eaten through, conclusions in regard to the quality of the material being drawn from the number of holes. This style of etching is not employed here but has been replaced by etching with copper ammonium chloride solution (12 g in 100 c. c. of water) the time employed being from 1 to 3 minutes. This etching fluid is chosen because it does not

cause any holes and so enables any want of continuity in the material to be recognised, while a microscopic examination is also employed in addition to the macroscopic examination which can be done when this etching fluid is employed.

Every microscopic examination is preceded in this institute by a macroscopic examination and it is in this way that the valuable information which matallography has provided, has been obtained.

In the course of these experiments we have come to the conclusion that in the present position of science it is not permissible to draw general conclusions in regard to the suitability of a material for any particular purpose simply from the extent to which it is attacked on etching with hydrochloric acid or the extent to which it is coloured to a greater or less extent on etching with copper ammonium chloride unless further indications are present. The matter requires care and the results obtained are not yet suitable for incorporation in contract specifications. The differences in tenacity in the different zones in a material do not always correspond with the extent to which it is attacked in particular zones by etching with hydrochloric acid or to the extent to which it is darcened by etching with copper ammonium chloride. It is not to be expected that the power of resistance to chemical action and mechanical forces are always similar.

4. The Phoenix Company for Mining and Metallurgy, C. Reinhardt, Laar near Ruhrort, Head Chemist.

a) I am acquainted with the fact that macroscopic examination of iron is carried out in the Rhine Westphalian and Lorraine iron industry.

b) Partially for scientific objects but mainly for the purpose of controlling the products produced in the works themselves or provided from some other sources.

c) The results give in the shortest possible time an idea of the constitution of the material as regards its strength, grain, equality, homogeneity, &c.

d) . . . . . .

e) There are a number of publications dealing with etched tests, e. g. »Stahl und Eisen« and the »Zeitschrift für das deutsche Eisen-Hüttenwesen«, Düsseldorf.

5. D. Spannagel, General Manager, Düsseldorf.

a) Yes.

b) Tests are made in a variety of German works on a large scale for the purpose of providing data in regard to the quality of iron and steel and the various iron and steel works have organised special departments for the purpose. The consumers do not trouble themselves much with such methods of testing.

c) In a large work which has been under my supervision for a long term of years a large number of such tests is carried out daily in order to obtain data in regard to the effects on edgings and piping and liquation. The etched tests provide the practised eye with a wonderfully clear view of the structure of the material and in combination with Smith's tests and tests for hardness enable the quality to be judged of with accuracy.

d) No, or only to very small extent (see the model specifications for iron and steel contracts drawn up by the Union of German iron manufacturers, Düsseldorf 1901).

I regard contract conditions of this kind as unsuitable because persons who have not had the necessary practice or experience may easily come to incorrect conclusions.

e) See «Stahl und Eisen» 1897 on piping in ingots by Ruhfus.

S. and E. 1896, p. 3 a piping phenomenon in hardened steel A. Ledebur.

S. and E. 1900, p. 774, recent progress in the manufacture of ingot metal by Fritz Lürmann, jr.

If I do not make a mistake, I believe that Ledebur's handbook of the metallurgy of iron which I have not at present by me, gives data regarding etching tests.

6. Professor H. Le Chatelier, Paris.

I have no data for answering these questions, but understand that the subject has been studied in France by Frémont.

7. The Railways of the East of France, Head Engineer, L. Salamon, Paris.

a) We make macroscopic examinations.
b) They have been employed in laboratories for scientific purposes.
c) We have not hitherto drawn any practical conclusions from such examinations.
d) Etching tests are not adopted in our contract specifications.
e) This subject has been dealt with in various publications principally by Osmond.

8. The General Management of the Norwegian State Railways, Divisional Engineer S. A. Lund, Christiania.

a) Etching tests are carried out by the General Management of the Norwegian Railways in the case of rails when fractures have occurred and then as close as possible to the spot at which the fracture has taken place. The etched surfaces are photographed and introduced into a treatise giving the weight of the rail, the name of the line, when the fracture took place, and so on.

Tests are only carried out with steel rails and not with iron rails.

b) No tests in addition to the above are carried out for scientific purposes. Such tests are sometimes made in supplying rails.
c) Definite results cannot be deduced from the tests above referred to; they commenced in March 1900.
d) Macroscopic examinations have not been introduced into any specification.
e) No, there is nothing of the sort in our country.

9. Technological Museum, Professor B. Kirsch, Vienna.

a) I believe so.
b) Both, but seldom.
c) I am not acquainted with these.
d) Also unknown to me.
e) No.

10. The private Austro-Hungarian State Railway, the Mining and Metallurgical Manager, A. Martinek, Buda-Pesth.

In reply to the questions directed to us, we would remark that etching tests are not carried out on our works.

11. The Royal Hungarian State Railways, Ministerialrat J. Geduly, Buda-Pesth.

a) Etching tests are carried out by the Royal Hungarian State Railways both for new and old rails.

b) Etching tests are conducted by us as purchasers of new rails on our works. Old rails are tested in this way when fractures occur or unusual wear has taken place. The tests are carried out in our technical laboratory.

c) Macroscopic tests are practically confined to Bessemer steel rails. In some cases fish-plates have also been etched.

I. New rails and fish-plates.

Of 56 rail sections etched, 28 corresponded to the heads of the ingots and 28 to the bottom. Of these 54 shewed pure steel and 2 impure steel. In some places want of continuity was observable. Of 10 fish-plates 8 shewed pure and 2 very impure ingot metal.

II. Old rails.

Etched sections of 47 different rails (old) shew in 19 cases pure steel and 28 cases very impure steel. The result shewn in figure 1 shews the etched section of a pointrail which split into 8 pieces under the Orient Express without any accident resulting.

Of rails that have worn down very much in a short time 13 of which were examined, 3 rails consisted of very impure material. Fig. 2 illustrates the etched section of such a rail from a tunnel.

e) Etching tests have not as yet been included in our specifications.

d) Not known.

Fig. 1.

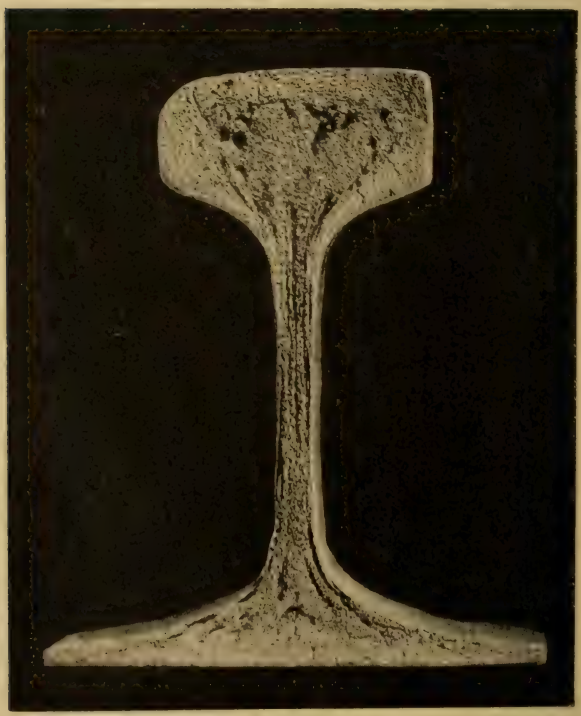

Fig. 2.

12. Royal Hungarian State Railways. Minister A. Czekelius, Buda-Pesth.

Ministerial Councillor Mr. Banovits, Hungarian representative of the International Union for testing materials for technical purposes, handed me a list of questions in regard to the tests that have hitherto been carried out for the macroscopic examination of iron with the request I should forward the answers directly to the President of the Union.

I accordingly have the honour to inform your Excellency that the macroscopic examination of iron has not in our case gone beyond the scientific stage of laboratory practice.

As far as practical uses are concerned the macroscopic examination of iron officially has not been adopted as a basis for contract specifications, the results not being looked upon as suitable for such a purpose.

I am avare that Prof. Alexander Retjö of the Buda-Pesth Technical High School has carried out such researches and that he will certainly be willing to place his results at the disposal of the Association.

13. Central Management Direction of the Royal Hungarian State Railways, the General Manager B. Veith, Buda-Pesth.

a) Macroscopic examinations (by etching) are occasionally employed in our works but only for the purposes of general control, for the purpose of ascertaining the homogeneity or the presence of flaws in the material.
b) As far as we know, such tests are not carried out in laboratories for scientific purposes and by producers.
c) General results have not been reached as the tests have not been directed to this end.
d) They have not been introduced.
e) In the technical publication: «A magyar mérnök-és épitészegylet közlönye,» Jahrgang 1899, Band XXXIII, Heft 6. Prof. Rejtö Sándor: «Szerkezeti anyagok mechanikai sajátságainak megállapitása» and in the publication «Bányászati és kohászati layok» 1903, No. 12. Prof. Faller Károly: «Tanulmányok a metallografia terén.»

14. The Institute for testing materials of the Swiss Polytechnic at Zurich, Prof. F. Schüle.

a) About ten years ago etching tests for iron were introduced into Switzerland by Prof. Tetmajer. These were employed on many occasions in the Federal Institute for testing materials and also in some Swiss works.

b) Enquiry has shewn that some large works employ etching tests for controlling their production. For instance, the Company of L. von Rohl at Gerlafingen and the Swiss locomotive and machine works at Winterthur. The tests of the Zurich institute are carried out both for scientific purposes and at the request of producers and consumers.

c) The etched tests have shewn themselves to be a very valuable method of research and in the technicological examination of iron enable local flaws, want of homogeneity in the material, piping and blow holes and grains of slag to be found by simple means. Cracks and blow-holes become apparent, even before the etching, in the polished surfaces prepared for that purpose.

As examples we would quote the following:

α) The etching tests shewed the causes of abnormally rapid wear of rails due to rolled out gas bubbles near the bearing surface.

β) They enable a fracture of a connecting rod to be shewn to be due to an imperfect weld.

γ) To enable the nature of the punching in boiler plates and the extension of the cracks in stampings to be made visible.

δ) They enable the frangibility of rails to be traced to either the top or bottom of the ingot.

ε) Etching tests enable the manner in which the head of a bolt was manufactured to be ascertained.

ζ) Experiments enable us to see whether wrought (puddled iron) is combined with mild steel or ingot metal in the same sample.

The macroscopic examination gives a complete picture of the long or cross section and is of great

value as a preparation for the microscopic examination of the grain, particularly for shewing what part should be so examined.

d) The macroscopical examination of iron has not hitherto been introduced in contracts in Switzerland.

e) Prof. Tetmajer has in the Swiss Bauzeitung Vol. 28, p. 130, treated of the manufacture of basic rails and in this treatise introduced photographic illustrations of the tests.

15. The General Management of the Swiss Railways in Berne.

a) Macroscopic examinations have only been employed in a few instances, particularly in the case of special fractures, in which scaling and blow-holes were visible, for the purpose of ascertaining the general structure of the rails. In the same way similar examinations have been made in the case of suspicious looking bridge-girders.

b) Some tests have been made by private railway companies at the Federal Institute in Zurich and the Institution has issued reports about them.

c) Conclusions cannot be drawn from the tests made.

d) In regard to the macroscopic tests nothing has been included in the contract specifications of the year 1893. We would of course have no objection to introducing conditions into the specifications based on such tests.

e) As far as we are aware papers on the subject by Prof. Tetmajer appeared in the Swiss Bauzeitung; we are not acquainted with any other treatises on the subject.

16. The Pennsylvania Railway, C. B. Dudley, Altoona.

a) Yes, such tests are frequently made by various persons more frequently with rails than with other objects.

b) We believe that most of these tests are made for purposes of study. They are also made by producers and consumers, but the existing want of certainty on the question of what it is that is really dissolved when the surface is etched, renders the results doubtful.

c) We are unable to say that any practical result has been obtained, except perhaps that the absence of homogeneity in iron or steel can be proved.

d) We were not aware that macroscopic tests have been included in specifications. We have seen none such.

e) Much has been written in the publications of various societies, particularly in the proceedings of the American Institute of Mining Engineers and also at times in other scientific publications, but it would be very difficult to find the exact references to them.

17. Pennsylvania Railway, P. Kreuzpointner, Altoona.

a) So far as I am aware the study of wrought iron and ingot metal by means of macroscopic tests is not extensively carried on either by producers or consumers. Five works and three railways to my knowledge study the structure of ingot metal for axles, crank shafts and tyres systematically macroscopically, for the purpose of ascertaining the structure and homogeneity of the material. In one metallurgical works this study was commenced in 1887, in another in 1889, and in three works in 1890 and 1891. Since the study of the microscopic structure of materials in the United States has become so generally adopted, more attention has been displayed upon macroscopic studies than formerly. I myself began this study in 1884. Macroscopic examination of wrought iron was less generally employed than that of ingot metal. I began the study of the macroscopic structure of wrought iron in 1883 and conducted the same on a very extended scale in the testing room of the Pennsylvania Railway where I still carry it on, the object aimed at being the examination of piled iron and welded material. I am not aware that any manufacturer has systematically carried on the macroscopic study of wrought iron or that any do so at the present date, in exceptional circumstances.

b) I am not aware that laboratories carry on the macroscopic examination of wrought iron and ingot metal. Probably such

experiments are conducted in the laboratories of one or other of the high schools without the results being published. As far as I know only a few manufacturers and consumers adopt it.

c) The results obtained by the macroscopic examination of wrought iron and ingot metal are thoroughly satisfactory and practically valuable.

d) I am acquainted with one class of material on which the results of macroscopic examination have been included in contract specifications. This is the wrought (or puddled) iron tubes for locomotive boilers. In the contract specifications for these tubes the railways proscribe that if the inspector has any doubt about the quality of the material — whether iron or steel (ingot metal) — he should test it by etching. In the cases in which the consumers have included in the specification that steel scrap (ingot metal) must not be used for piling with puddled iron, the inspector is often compelled to adopt the etching test to see whether steel scrap has really been employed.

e) I am not acquainted with any publications dealing with the subjects of macroscopic tests. Howe in his Metallurgy of steel only refers to it very briefly.

B. Historic development.

Etching iron in order to display the texture has been long in use, particularly in the manufacture of weapons. Damascus steel and damascened wares are universally known.

A similar phenomenon is also familiar in the case of bells, handles and other wrought iron objects exposed to the action of atmospheric influences, namely that the macroscopic structure is brought by these influences prominently into view.

Though known for many hundreds of years and used for decorative purposes, it is only in the last few decades that etching has been employed for the purpose of testing, and only in the last decades has it been shewn that the process is applicable or capable of providing valuable data in regard to the quality of ingot metal.

In the «Berg- und Hüttenmännische Zeitung» Leipzig 1872, is a communication from Mr. Van Ruth stating that he has discovered a method of testing the quality of iron by treating the samples with acids for the purpose of disclosing any imperfect portions of the texture. It is probable that this is the first occasion in which etching has been employed for determining the quality of iron.

A large number of etched sections of iron were exhibited at the Vienna exhibition in 1873.

The first publication in Austria in which etching is recommended for testing purposes probably dates from the year 1873. This is a communication to the journal of the German Polytechnic Union in Prague by Councillor Prof. Kick. This work contains a number of illustrations of macroscopic structure of welded material from a Bessemer ingot and a Bessemer rolled sheet from Neuberg. The author described etching as a valuable method of testing for producers and consumers of iron.

In Germany etching has first been recommended by Prof. Bruno Kerl in his work, Outlines of Metallurgy, 1875. It is pointed out that the brighter and darker surfaces of the etched material correspond with the degree of carburisation and the amount of foreign constituents.

In the year 1878 followed the great work «Concerning railway rails» by Councillor Prof. A. R. von Kerpely, professor in the Mining School of Chemnitz. In this work in addition, to a number of etched sections of puddled iron there are introduced etched sections of rails and shafts of ingot metal. Kerpely attributes the inequalities in the etched tests in the case of ingot metal to inclusions of slag and difference in the degree of hardness, owing to the fact that slag and soft iron are more rapidly attacked by acids and comes to the interesting conclusion: «We see once again what valuable services the etching test is capable of rendering in most instances and particularly in the case of deliveries of carriage axles.»

After the appearance of these publications concerning the etching process and its employment for testing the quality of iron a certain amount of attention was bestowed on the subject by

some manufacturers and consumers in Austria and other countries. Irregularities in the etched figures were attributed to variations in rolling and forging.

In 1882 Privy Councillor Wedding stated to the «Verein für Eisenbahnkunde» that the Nordhausen Erfurt Railway introduced etching of suitably prepared sections as a qualitative test in their contract specifications for rails. The passage in the said specification is as follows: «The etched surfaces must neither be unequally hard nor show soft places or veins and must show no holes particularly at the edges of the sections.»

Of further works on the subject which have appeared in Germany in regard to etching tests specially to be noted are the following, Ledebur 1886 to 1889, S. Stein 1887, Wedding 1891, Martens 1892 and 1894. In all these publications which have appeared in «Stahl und Eisen», a number of interesting observations are introduced. Specially noteworthy is the description by Ledebur of the formation of a star-shaped nucleus in forged drawn bars of ingot metal (fig. 3), this nucleus being more powerfully attacked

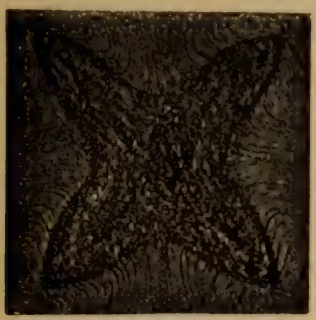

Fig. 3.

by acids than the parts surrounding it. Of great importance also was the observation by Wedding in sections of rail heads of a close grained sickle-shaped material (fig. 4) enclosing a less compact nucleus and itself enclosed by a similar layer of material. Martins in his paper on «Piping and iron and steel castings» at the Engineers' Congress in Chicago in 1893 two cases were discussed in which etching with acids gave good results. Eccles

etched 40 parts of plates (Journal of the Iron and Steel Institute 1888 No. 1) in which the central portion was completely eaten out by the acid. A. A. Stevenson and R. Kent in examining tyres employed etching as a preliminary test before making chemical analysis, the etching test being used to indicating those spots to be analysed.

In 1896 the important researches of the Kaiser Ferdinands Northern Railway were published*) which were mainly concerned with rail steel. In this work attention is drawn to the formation at the edge zone and central zone material and it is pointed out that the phenomena shewn in the etched sections point to the effect of separate qualities in the parts of the material in question and that they enable the causes of the fractures to be quite simply explained.

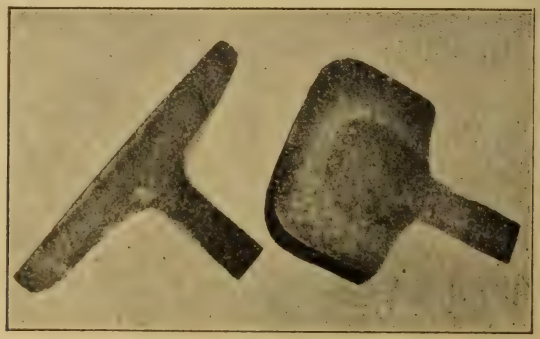

Fig. 4.

The observations made by the Northern Railway have been confirmed by Prof. Tetmajer in his extensive work on the Metamorphoses in Basic Rail Steel, and methods of testing steel rails», which appeared in vol. 28 of the Swiss „Bauzeitung" in the year 1896. The occurence of edge and central zone formations is proved in this work of Prof. Tetmajer and the influence produced by foreign inclusions and gas bubbles is discussed. Prof. Tetmajer regards the etching test as a sort of prognosis in regard to the behaviour of rails in tests and considers it in consequence as a process suited for rendering the ordinary

*) A. v. Dormus, «Zeitschrift des Österr. Ing.- und Arch.-Vereines» 1896.

tests complete. His first experiments were conducted with dilute hydrochloric acid, but the desire to obtain a rapid method which can be used in the same sort of way as ordinary fracture tests in the testing machine gave rise to the employment of iodine as the etching material. This process gives much more distinct figures of the rough structure of the material since the impurities in the steel are not dissolved and the differently carburised portions are rendered noticeable by differences in the shade of colour imparted to them.

Tetmajer states that the process has been very fully developed in a masterly way by the head chemist Reinhardt of the Phoenix works, in Laar near Ruhrort. The Phoenix works exhibited a number of etched sections of compressed and uncompressed ingot metal at the Düsseldorf exhibition of 1902.

At the Stockholm Congress of the Union in 1896, the Reporter discussed at considerable length certain relationships between the macroscopic granular structure of rail steel and its durability when in use. The formation of edge and central zone structure is also discussed by Ruhfus in a paper published in 1897 in «Stahl und Eisen», «Concerning liquation in Ingot Metal». Ruhfus gives an exhaustive description of the process of liquation and shews by etched ingot sections taken from the head or upper end what the effect of the temperature of the different «blows»

Fig. 5.

is on the thickness of the edged zone. Thus the section in fig. 5 corresponds to a «hot blow»; fig. 6 a «blow» of medium temperature and fig. 7 a «cold blow». The edge zone is pure or only slightly impure.

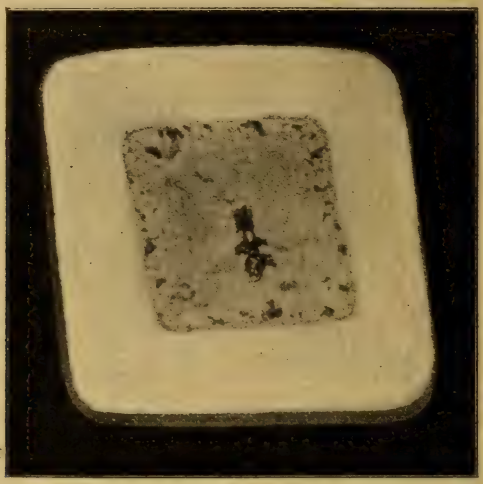

Fig. 6.

In the years 1898, 1899 and 1900 further researches of the Northern Railway were published (A. v. Dormus, «Zeitschrift des Österr. Ing.- und Arch.-Vereines», 1898, 1899 and 1900) dealing

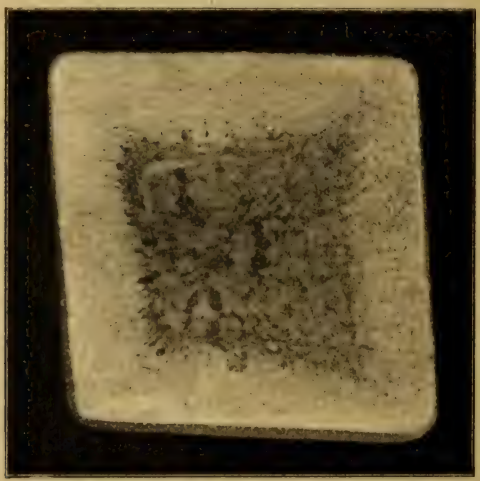

Fig. 7.

Fig. 8.

with the macroscopic structure of ingot metal and the durability of this material in use. A new observation is mentioned namely the tendency to redshortness of the more impure nucleus steel which results in interruptions in the continuity of the material when forged at a red heat, the results occasionally being fracture when put into use. Some examples from practice are introduced to prove the correctness of these statements.

In the year 1899 appeared in the «Journal for Building Materials» a paper entitled «Do the tests for steel rails fulfil their purpose?» — an extensive work by Engineer D. Miller in which a number of etched sections of rails are reproduced. The writer recognises the value of the etching test and mentions a number of cases in which want of continuity in rail heads gave rise to fracture. Fig. 8 gives the surface of fracture and fig. 9 the etched profile of such a rail.

Fig. 9.

Prof. Heyn in »Stahl und Eisen« 1899 in a paper on the fine grain structure of iron, describes the appearance of the edge and central zones in shafting.

These shewed quite considerable impurities in the central zones and all of them had been fractured under ordinary working conditions after being only a short time in work without visible cause. Prof. Heyn is of opinion that liquation should be regarded as playing an important rôle in determing the quality of the material. He etches with copper ammonium chloride, and not with hydrochloric acid, because with the former the same data are obtained as with the latter while the fine grain structure remains clearly visible.

Prof. Martens in »Stahl und Eisen« 1900, is of opinion that the etching test may be regarded as a valuable auxiliary means for judging of the probable behaviour of rails under working conditions, but that great caution must be employed in drawing any general conclusions from etching tests. In the royal Technical Testing Institute in Berlin etching with acids has been given up and copper ammonium chloride is at present employed, by means of which excellent results are obtained in a short time.

At the Paris Material Congress in 1900 a paper on the subject of rail steel was read by Engineer Frémont in which a parallel was drawn between etching and drop flexure tests. Frémont speaks unfavourably of the etching process as a means for determining the quality of ingot metal.

Engineer Fritz Lürman jr. in »Stahl und Eisen« for 1900 and more recently the General Manager August Spannagel in Düsseldorf described the etching test as a valuable means for steel works to control the quality of their output.

A complete determination of the relationships obtaining between the macroscopic structure and the manner in which the material behaves in practice has been supplied by one of the reporters to the Buda-Pesth Congress for 1901. The fractures of rails, axles and other parts are described therein and explanations given. The explanation of the causes to which a number of small hairlike fractures are due which appear occasionally at the foot-edges of rails is a subject which requires

further study. To this question belongs the occurrence of a purer zone between the edge and central portions of the steel and also the occurrence of redshortness which has occasionally been remarked and has been occasionally noticeable even after ingot metal has passed through the rolls and been cut by the shears. The relatively frequent and advantageous character of the etching tests are borne witness to by these communications with the replies to the questions in the circular letter, where similar evidence is provided by the most recent publications dealing with the same subject. On the first December 1904 Samuel A. Voulcain of the Franklin Institute in Philadelphia reported to »Stahl und Eisen« 1905 in regard to some etching experiments which might be employed with advantage for testing the quality of ingot metal employed in the manufacture of tyres and wheels. In the report of the engineer P. H. Dudley, Inspector of the New-York Central & Hudson River Railroad in 1905 which was placed before the International Railway Congress the statement is made that various American Railways have employed etching tests satisfactorily for the purpose of finding out to what causes abnormal destruction of rails is due. Mr. R Job, Chemist of the Philadelphia & Reading Railway Co. has written on the same subject and placed his paper before the American Society for testing materials.

The knowledge which we possess regarding the nature of the etching test has ben increased in recent years by the study of the chemical behaviour of ingot metal.

As regards the influence of a percentage of copper in effecting the tendency of ingot metal to rust, attention is drawn to the paper of F. A. Williams in the Mining Journal, LXX p. 667, and to the Austrian Zeitschrift for Mining and Metallurgy 1904, page 559. Similar tests have been continued by Bergrath L. Schneider in Austria. They refer to the influence of copper, sulphur and phosphorus on the chemical behaviour of ingot metal.

The study of the microscopic structure of iron has, considering the great importance attributable to it, been carried out through a long period of years, and it is to be hoped that we shall see during the next few years still outstanding questions on this important subject satisfactorily answered.

C. Present Position.

The object of etching with acids or other reagents, is to obtain a glance into the internal structure of the iron in order to form an opinion regarding its probable behaviour in use. The internal structure is very varying according as puddled iron or ingot metal is being subjected to examination and provides such characteristic appearance in either case as to enable these important species of iron to be readily distinguished from one another. Etching is accordingly employed largely for the purpose of distinguishing them, more particularly as it is the only method that exists of doing so.

Ingot metal. The characteristics of the coarse structure are formed in the moulds by the iron passing from the liquid to the solid state. The solidification commences at the edges and round the sides of the mould and progresses thence towards the centre of the ingot until this portion has assumed a pasty consistency and also at almost the same instant solidifies. In combination with this phenomenon the liquation products are gradually forced towards the centre, while they rise to the top very largely as the result of the liberation of the occluded gases. The ingot accordingly consists of a purer external zone and a central zone, the latter containing the liquation products; the surface which separates these two portions of material is called the solidification surface, the lines of strain across it, which can be made sectionally visible are termed the solidification lines. On these often round bubbles occur which sometimes form a complete circle; in the edge zone material occur, at right angles to the solidification surface, the edge bubbles, often in regular honeycomb fashion. Peculiar to the central zone are the round bubbles and the contraction spaces known as pipings. All these phenomena which give rise the general macroscopic structure of the material occur more or less completely developed though at times some of them are completely absent. The most important influence determining which of these phenomena shall be most prominent, and which less so, is the procedure at the end of the manufacture of the steel (in the converter or furnace), the chemical composition, the temperature of the steel when cast, the method of casting adopted and the conditions under which cooling takes place.

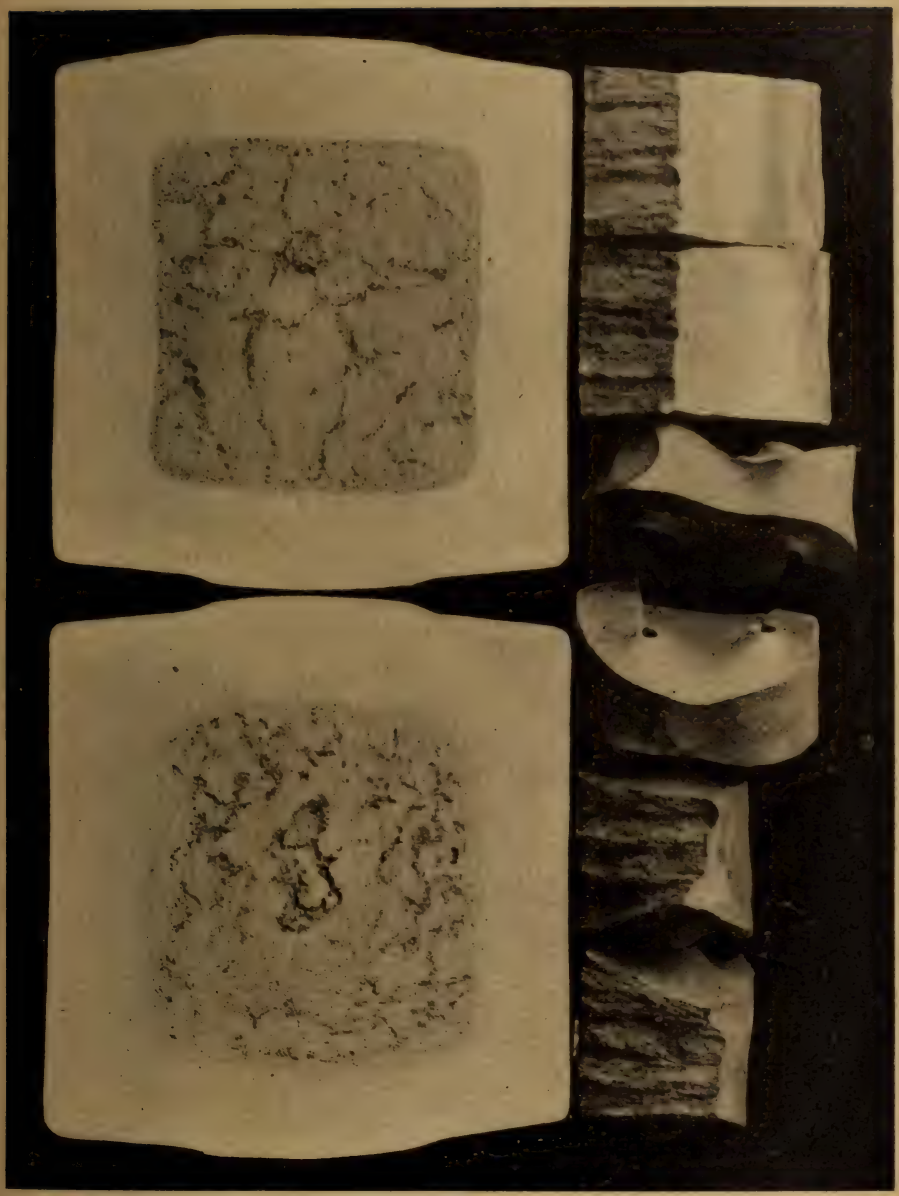

Fig. 10.

When the ingots are broken or cut the cavities in the material become apparent, whereas the position of the liquation products and their distribution in the central zone can only be rendered visible by the employment of chemical reagents such as etching materials. By rolling, forging and moulding the cavities which exist in the ingots are mostly closed up; they escape observation accordingly unless they happen to contain large inclusions of slag, they can however be rendered visible by the employment of the same means as are used to disclose liquation products namely, (etching materials). This is due to the fact that the walls of the cavities are coated with liquation products and the cavities accordingly present the appearance in the forged metal of fine liquation veins. It is possible therefore to determine the formation of the internal structure of the ingot metal in the final product in a simple manner. It should here be observed that the coarse structure (macroscopic structure) as opposed to the fine grain structure (microscopic structure) only undergoes an alteration in form by mechanical treatment such as rolling, hammering, or hydraulic pressing, by being displaced longitudinally. In material that has gone through the rolling mill, therefore, it will in general be sufficient to test two sections, from portions corresponding one to the top and one to the foot of the original ingot.

The preparation of the section of which the texture is to be examined, is as follows: The specimens are first of all polished as smoothly as possible though they must not be polished with steel instruments. For examining the coarse grain or macroscopic structure rubbing smooth with emery suffices. When the fine grain or microscopic structure is to be tested, the samples must be polished carefully with the finest emery and rouge.

The etching fluids employed are preferably:

1. Dilute hydrochloric acid, nitric acid and sulphuric acid, in proportions varying up to 30% of acid.

2. The solution of 20 gms. of iodine and 30 gms. of iodide of potash with 1000 gms. of water (Tetmajer).

3. Hydrochloric acid diluted with alcohol in mixtures up to 30% acid (Martens & Heyn).

4. 10% solution of copper ammonium chloride (Martens & Heyn).

5. 10% solution of ferric chloride.

Fig. 11.

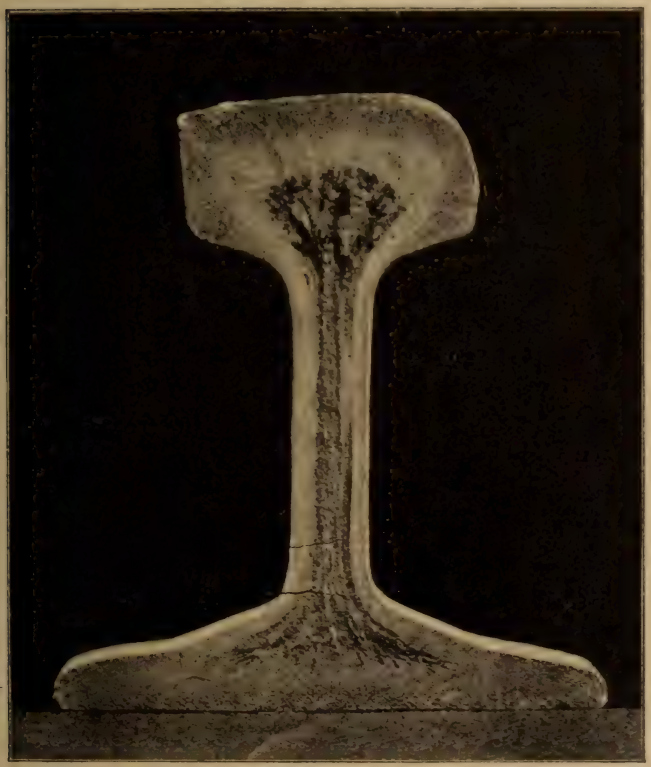

Fig. 12.

The effect of these reagents depends upon the fact that they attack irons of different chemical composition with different degrees of energy. As regards the variable solubility of ingot metal and its chemical properties the researches of J. H. Williams and Bergrath Schneider already referred to have shewn that an effect is produced by *Cu*, *S* and *P*. The most important effect is produced by copper and tin which diminish the chemical activity of the iron. As example may be cited the fact that the solubility of iron free from copper and an iron containing 0·15% *Cu* under otherwise similar conditions are as 25 to 1. This not infrequent constituent of ingot metal, however, does not liquate and consequently exercises an influence on the macroscopic determination of texture only in so far as it renders it necessary to vary the period of exposure to the reagents according to the percentage of copper present.

Of much more importance is the influence which the elements *C*, *S* and *P* exercise, as they are the substances which have the greatest tendency to liquate out. Their effect is unequal as the chemical activity of the iron diminishes with the amount of carbon it contains. As the amount of sulphur and phosphorus however, increase, its chemical activity is also increased and the effect of the latter element is more pronounced than in the case of carbon even though their tendency to liquate out is still greater. In addition to these constituents of ingot metal, manganese and silicon are to be considered, on the effects of which on the chemical activity of iron no data have been obtained. Manganese liquates hardly at all and like copper can therefore only effect a variation in the time needed for exposure to the reagents. The case of silicon which liquates notably, is different. According to the experiments that have been made, this element appears to increase the solubility of the iron.

In addition to *C*, *Si*, *P*, *S*, *Mn* and *Cu* many other substances occur in admixture with ingot metal prepared in the ordinary way. Even when we leave out of consideration gases and inclusions of slag which are not of much importance, the effect they produce, though only present in small quantities upon the solubility of iron and in consequence of the appearance of etched samples may nevertheless be at times noticeable. To determine what the effects are would require an enormous amount of labour.

Among the elements which affect the coarse structure of the metal and have accordingly the greatest influence on determining the appearance it presents after etching, must be mentioned the elements phosphorus and sulphur, as they are of constant occurrence in iron and as they have the greatest tendency to liquate out and as they are the most effective in increasing the chemical activity. The latter of these two elements it must be admitted operates to prevent the separation of the carbon content by means

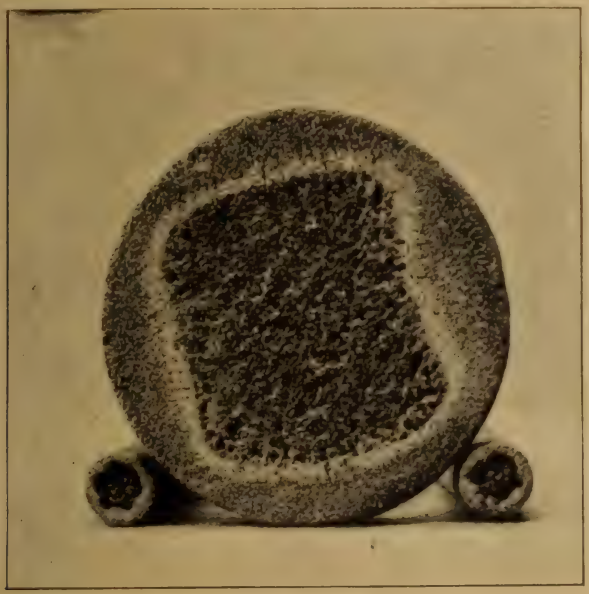

Fig. 13.

of liquation, but this can only be the case when the said elements are present in very small quantities and consequently did not liquate out much while the carbon at the same time occurs in large amounts.

By employing acids as etching materials, the etching is usually carried deeper and an exposure of hours or days is frequently necessary. The most powerfully etched portions of a section appear also to be more darkly coloured. In tests for the macroscopic structure which should also enable the microscopic structure to be examined other chemical reagents besides acids

are frequently employed. The time of exposure in this case amounts only to some minutes and the different portions of the test section appear differently coloured. Both methods of testing have their advantages and disadvantages.

Before introducing the test pieces into the etching fluid they must be previously freed from grease by means of alcohol or alcoholic solutions of caustic soda or caustic potash. Where a slight amount of etching only is to be carried out the surface cleaned in this way should be dried with ether. After careful etching the test surfaces should be carefully cleaned in water, more deeply etched surfaces being allowed to remain for a long period in the water so as to fully ged rid of the etching fluid. After washing the test pieces should be dried lightly with a cloth. In the case of deeper etchings the water should be removed by means of alcohol, the alcohol by means of ether. If the test pieces are to be preserved they should be protected by a thin layer of Damar lac dissolved in toluene. In many cases a suitable effect is produced by a 5% solution of waterglass in which the test pieces are permitted to remain for some hours and the solution wiped off lightly with a cloth. Protecting layers of this kind are of special importance where acid etching has been employed as otherwise the test pieces rust very rapidly.

The test pieces illustrated in reference to the present report were almost without exception etched with 30% hydrochloric acid and protected with a layer of water glass. Figure 10 shews the two samples treated in this manner of a section from an ingot in which the edge zone and central zone formation is very easily observable. The line of solidification is not always marked sharply, as can be seen from fig. 9. The amount of surface covered by the edge and central zone can vary considerably. It is mainly influenced by the temperature of the steel at the time of casting, as already observed in reference to figures 5, 6 and 7. The edge zone can under certain circumstances, as in (fig. 11) diminish to a minimum In any case, it should be borne in mind that the outsides of ingots tend to burn off in the mill furnace which is one of the reasons why the edge zone in the specimens that have gone through the rolls is smaller than it should be. In many etched sections (fig. 12) the edge zone consists of two parts — of an outer somewhat impure zone and of a purer

portion connecting this with a central zone. This phenomenon is due to the circumstance that the outer layer has solidified very rapidly, from which cause the impurities have only been able to separate out imperfectly, whereas in the other the edge zone has been formed more slowly and the impurities have accordingly been

Fig. 14.

more completely liquated out. These purer edge zone portions are specially met with where large ingots are rolled down into a small section. The larger section in fig. 13 corresponds to a round bar in which the ratio of ingot to finished section are represented by 350/1. The interior edge zone portion appears as a fine ring in the etched section. In the same figure too the etched section of 5 mm rolled wire is shewn, in the production of which the original ingot was rolled down to 0.000125 of its original section. The

great mechanical work expended in effecting this to which the material had to submit has scarcely had any effect in altering the sensitiveness of the central zone to the attacks of the acids.

The distribution of the impurities in the central zone is seldom equable. In general accumulations occur along the line of solidification (fig. 14). Centres of liquation also occur, however, of irregular distribution and small size (fig. 26).

The various differences in qualities of edge and central zone material as far as experiments have yet been made, would appear to be essentially as follows:

The edge zone material is in the great majority of instances of equal tensile strength and toughness throughout the ingot. Variations in this respect occur mostly when the extension of the central zone is small. The edge zone material is in general free from any tendency to redshortness. Two samples shewn in figure 10 of bars from edge zone material which were tested from this point of view, shew that they are composed of a thoroughly sound ingot metal.

The central zone material is in general of greater strength and less ductility than the edge zone material, the distinction being less marked at the bottom of the ingot and sometimes wholly disappearing there, though increasing towards the top of the ingot. A special feature of the central zone material is the occasional prevalence of redshortness, a characteristic the presence of which can be proved when only comparatively small amounts of impurities are present, and where local accumulations of the latter are specially marked. The two outside rods in fig. 10 correspond to the central zone material and shew marked redshortness.

Many etched cross sections which correspond to the tops of the ingots shew little or no difference between the edge or central zone. Material of this kind is pure and mostly free from redshortness. It can, however, display very marked variations as regards its capabilities of resistance to tensile strain according as the test pieces are taken from the edge or central zones. Phenomena of this kind occur in a marked degree when the recarburisation of the steel in the furnace has been inadequate in which cases the recarburising material (high percentage pig, spiegel, &c.) has not been fully dissolved.

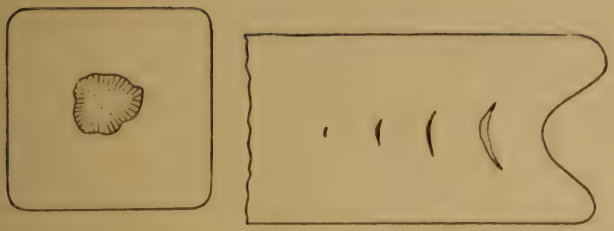

Fig. 15.

Any redshortness present is frequently shewn when the ingots are cut up after going through the cogging mill, by more or less large portions being dragged from the centre as shewn in fig. 15. By further rolling down cracks appeared in the cooler finishing rolls as shewn in fig. 15.

In the etched ingot-iron profiles the occurrence of piping due to leaving the dead heads of the ingots too short and imper-

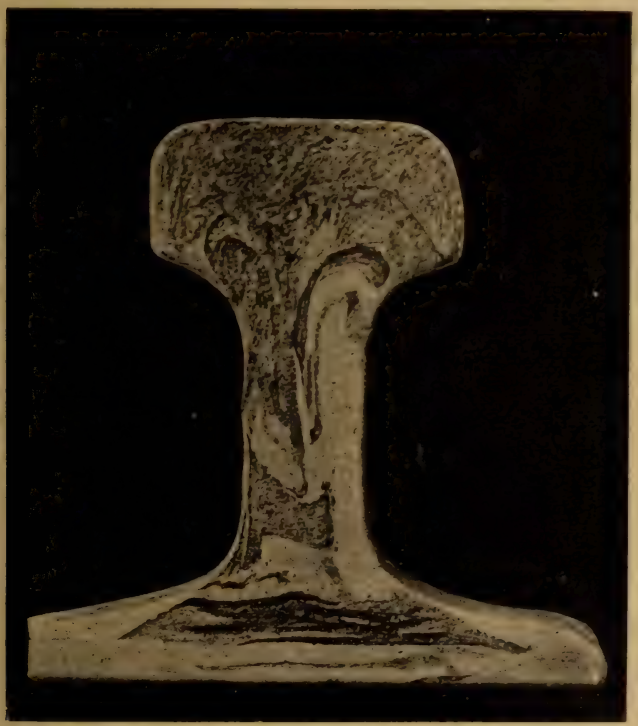

Fig. 16.

fect parts due to fissures are visible. It was owing to this circumstance that this etching test was incorporated into the contract specifications of the K. F. Northern railway. From each portion of each charge a section corresponding to the top of the ingot is etched.

The impurities of ingot metal affect its electrical conductivity.

Puddled iron. Etching enables the kind of piling employed, the extent to which the whole has been rendered homogeneous by welding and the quality of the iron used to be ascertained. Etching therefore, in combination with the usual mechanical test has accordingly proved to be a valuable means for determining the quality of «merchant» or puddled iron. The preliminary preparation of the pieces to be tested and the etching of them take place in the same way as in the case of ingot metal. Fig. 16 shews the etched cross section of a puddled iron rails; fig. 17 shewing a puddled steel rail.

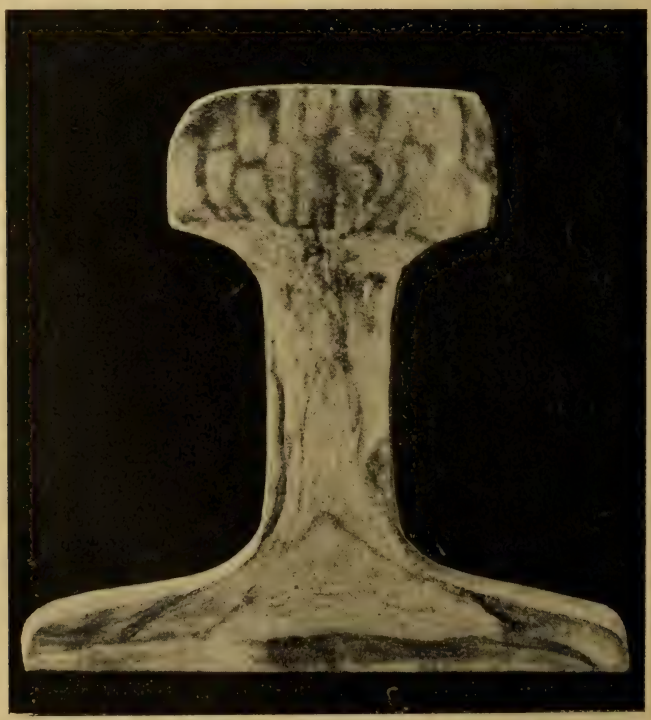

Fig. 17.

D. Influence of macroscopic structure of ingot metal and its importance in practice.

According to the experience at present at our disposal the redshortness of the central zone material would appear to be the cause of various failures of material owing to the impurer parts of the metal being liable on account of the presence of impurities,

Fig. 18.

Fig. 19.

to crack and tear when being forged at a dark red heat. Failures of this kind in the continuity of the material on a smaller or larger scale are the origins of fractures. Externally they are as a rule invisible and more dangerous the more sensitive the material is to injury. Discontinuity of the material of the kind described, was in a number of cases shewn to be the cause of rail fractures (and in some cases of splitting) which have actually given rise to accidents.

Fig. 20.

The etched rail section Fig. 18 was obtained from a rail which broke in use. The round rod on the left with a number of imperfect spots and the two rods on the right which were

Fig. 21.

examined for redshortness, were taken from the middle of the head parallel to the length of the rail. Figs. 19 and 20 correspond to the broken surfaces of two rails broken in use. Figs. 21 and 22 shew the corresponding etched section. In both cases the

Fig. 22.

fracture occurred at a spot where there was a regular collection of impurities. The originally formed fissures have in course of time increased in width and ultimately produced complete fracture.

The imperfect portions in the interior of the material due to redshortness have in many cases caused splitting of the rail heads (fig. 23) and scaling off of the substance (fig. 24). Rails with very fine hair fissures in the foot in many cases give etched figures like fig. 25, which comes from a rail of this kind. The

Fig. 23.

greater amount of impurities along the line of solidification, which in this case, occurs very near to the surface, was the case of these hair fissures, when the edge of the rail foot during rolling happened to attain the temperature at which redshortness displays itself. It is conceivable that ingots in which the distribution of the liquation products is as described are not suited for treatment in the rolls at reduced temperature as recently recommended.

Fig. 24.

Similar fracture phenomena as in the case of rails, also occur in the case of axles. Considering the greater reduction in section to which the material is subjected the occurrence of injuries as the result of redshortness is easier to explain than in the case of rails. The etched section of a carriage axle which broke in use (fig. 26) shews that the flaw which occasioned the fracture,

Fig. 25.

existed before the forging was begun and grew during the time the axle was in use, the cause being the local aggregation of liquation products. The illustration shews the fissures as they appeared before etching. The portion of the axle inside the wheel had also flaws which occurred in a direction at right angles to the length of the axle (fig 27) and these gave rise to the discovery of the fracture. Axle fractures of this kind which are to be traced to similar causes, occur not unfrequently.

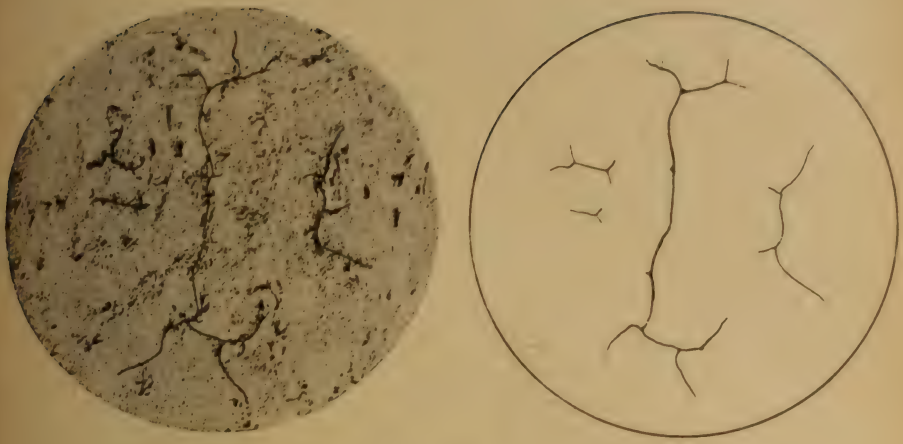

Fig. 26.

Many descriptions that have been given in the literature on the subject and illustrations of fracture, such as occur in the case of propeller shafts and other parts, lead us to suppose that they are due to similar causes to those which have just been described.

It must be pointed out with emphasis that the discontinuities in the material resulting from the presence of redshortness do

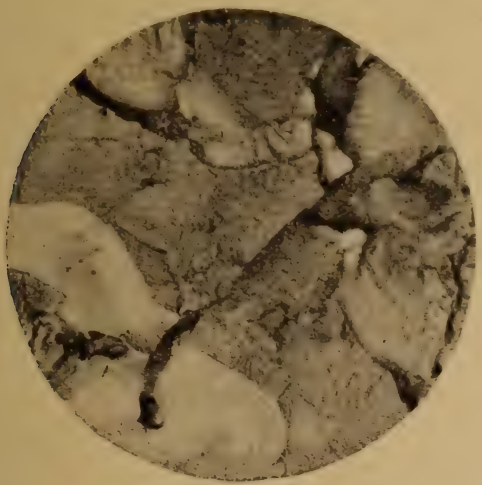

Fig. 27.

not always result in large surfaces. Only very slight and scarcely recognisable injuries are sufficient to cause a tendency to fracture when they occur in large number, the tendency to fracture manifesting itself when the material is exposed to heavy wear and tear. This gives rise to a slow and gradual widening of the injuries. Even quite normal wear and tear may give rise ultimately to complete fracture.

The abnormal heavy wearing down of rails may in many cases be traced to macroscopic structure.

After the fact has thus been pointed out that many fractures of material are to be traced to imperfect structure i. e. to great impurity of the ingot metal, it must on the other hand be observed that very frequently materials with very imperfect structure will withstand continuous work without injury and do excellent service. This apparently paradoxical phenomenon is to be explained by the fact that materials of this kind have been worked up in a way in which it is impossible for disturbance of the material to occur, that is to say they have been forged at higher temperatures and not at the temperature at which redshortness usually displays itself. Starting points for fracture have consequently not been formed. In many cases also the tendency to redshortness has been diminished by the well known expedient of increasing the percentage of manganese which the material contains.

The fact that materials of very defective structure under certain circumstances comport themselves well is the cause why less attention has very often been paid to the etching test than it deserves. It is also true that serious impurities do not now occur in ingot metal so often as formerly, but still they do occur and consequently a method of testing for their presence in materials is required. Material with small amount of impurities is always to be preferred, as we possess no means of deciding whether injuries to its structure due to a disposition to redshortness in the central zone material are actually present or not. While the products of one ingot which may have been rolled in a cooler condition than another may display them, they may fail to occur in the products obtained from other ingots of the same charge or «blow» which happened to be rolled at a higher temperature. A material which contains a large amount of impurity is therefore always to be regarded as of doubtful value.

What has been said above includes the most important work that has been carried out as regards the macroscopic examination of iron, and gives the most important remarks in reference to it of the highest authorities. Absolute completeness cannot be claimed; but this is not necessary in order to enable a competent view of the international results which have been obtained and an adequate estimate of the present position of the macroscopic examination of iron to be formed. The following resumé will adequately summarise the present condition of affairs.

E. Resumé.

1. Macroscopic examinations of iron have been carried out in Denmark, Germany, England, France, Norway, Austria, Hungary, Switzerland and the United States of America, but they have only found extensive employment in Germany, Austria, Hungary and Switzerland, and this method of testing has been principally developed in the latter named countries. In the U. S. A. it has, since the microscopic examination has awakened so much general interest, been generally forgotten. It was supposed that it was a very good method of distinguishing good from bad material, but the doubt which prevailed as to which portions of the material were dissolved in etching, left the question unsettled.

2. Macroscopic examinations of iron are frequently employed in laboratories for scientific purposes, and as an additional test for the purpose of deciding the causes of fractures and where there has been great wear and tear of material. Various consumers employ it in their own works for controlling the quality of the material for contracts. In the case of merchant iron it is employed to test the quality of the piling. In the testing institutes of Charlottenburg and Zurich the macroscopic structural tests is employed as a preliminary test in the microscopic examination of iron. In many German and some Austrian works the test is employed for controlling the works management. Many metallurgical laboratories have special departments for this purpose. It is generally recognised by the majority of authorities as of the greatest value for determining the quality of ingot metal. At the same time great practice is required to enable an adequate judgment to be formed.

3. Etching with acids enables the impurities to be detected: etching with iodine and copper ammonium chloride enables the more highly carburised portions to be detected as well. The appearance of the etched surfaces gives with a little practice the means for forming the judgment of how the material will behave in the testing machine in practical use as also of its electric conductivity. The most noticeable injurious quality in a material containing a large quantity of impurity is a tendency to redshortness, which under certain conditions gives rise to discontinuity of the material which may have very serious effects upon its practical life in use. Many fractures are to be traces to these phenomena.

4. The etching test is regarded by many authorities as not sufficiently developed to be included in specifications, as in its present state it does not provide numerical data that can be made use of and is very likely to lead if adopted by inexperienced inspectors to the rejection of material which ought to be accepted. The etching test is however to be recommended as the preliminary test for deciding from what portions to select the test rods.

According to the above reports the etching test has been incorporated in the rail contracts of the K. F. Northern railway to provide a control as to whether the ingots from which the blocks have been removed have had sufficient deadhead allowed. Such conditions have also been incorporated in American rail contracts to distinguish puddled iron from wrought iron and particularly in regard to the latter to ascertain whether steel scrap has been added.

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INTERNATIONAL ASSOCIATION FOR TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 37.

REPORT ON THE PROGRESS OF METALLOGRAPHY SINCE THE CONGRESS AT BUDAPEST 1901.

BY
F. OSMOND AND G. CARTAUD
PARIS.
TRANSLATED BY CH. CAPITO, LONDON.

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Report on the Progress of Metallography since the Congress at Budapest 1901.

By **F. Osmond & G. Cartaud.**

(Translated by Ch. Capito, London).

Since the last Congress of the »Association Internationale des Méthodes d'Essai«, the movement, mentioned by one of us at a conference during the Congress at Stockholm, for connecting more closely metallography and physical chemistry, has become more and more marked. One might say that the union of the two is now complete, and that the micrographical study of alloys has become one of the branches of physical chemistry, together with expansibility electrical resistance and all other physical properties. It assists in the most efficacious manner towards the solution of the general problem: to determine the conditions of existence (concentration, temperature, pressure, etc.) of each of the phases (solid, liquid and if it takes place, gaseous) which can be formed by two or more bodies associatêd.

It is natural that metallography should borrow the language used in general science of which it has become an auxiliary, and for this reason we have used the word »phase«. To give the explanation of the principles and terminology of physical chemistry is evidently beyond the compass of this report. We must however assume that our readers possess a fundamental knowledge. To those who have not followed closely the rapid flow of ideas and who are anxious for information, we would recom-

mend particularly an article by H. Le Chatelier[1]), a pamphlet by Prof. Heyn[2]) and a book by Prof. Howe[3]).

In the article of Le Chatelier, the phase rule is explained without the aid of the mathematics with which its originator, Professor J. W. Gibbs, has associated it. The general principles are specially explained and illustrated by some examples.

Prof. Heyn describes a series of examples extending gradually from the most simple to the most complicated; without making use of the terminology of the phase rule, he is nevertheless guided by its ideas, and without the reader perceiving it, gradually prepares him for it.

Prof. Howe follows a similar route, but goes steadily on till the end of his work. The phase rule thus appears, not as the fundamental axiom on which geometricians base their theorems, but as the natural consequence of experimental facts judiciously selected. The route followed is not that of the technical college, where the courses depend upon each other and are addressed to an uniformly prepared audience. It is probably more suited for the engineer who has already started practical life, and who is obliged to acquire slowly the new ideas which were not prepared for him in his younger days.

Neither must we forget Baron Jüptner von Johnstorf. One of the foremost, he has thoroughly understood the importance of the phase rule for metallurgy, and he has earnestly devoted himself to as application. His admirable book »Grundzüge der Siderologie (Outlines of Siderology), Leipzig, A. Felix, 1901« is the result of his conscientious efforts; all previous works are there carefully summed up and arranged in a new order.

By the aid of these masters, it will be comparatively easy to acquire information about the progress which has been made, and to read with advantage the most recent works, dealing with the problems of metallurgy by means of the phase rule.

[1]) La loi des phases, Revue générale des sciences pures et appliquées. X, 759, October 30th 1899.

[2]) Die Metallographie im Dienste der Hüttenkunde. Craz und Gerlach, Freiberg, 1903.

[3]) Iron, Steel and other Alloys, Sauveur and Whiting, Boston, 1903.

It would take up too much time to give a resumé of all the work which has been done during the last two and a half years in physical chemistry relative to alloys. We are bound to limit ourselves, in conformity with the title of this report, to those in which the microscope has been used. And yet we are not sure that our report is complete, notwithstanding the help of the excellent special review by Prof Sauveur, »The Metallographist« to fill up our programme. With regard to the Russian works especially, which are not accessible to us in their language, we have not received the information which we had asked for, and on which we had reckoned. We regret this all the more, as this report is to be presented at St. Petersburg; but we hope that the Russian metallographers will give the Congress the benefit of their researches.

Even in this limited region, it is impossible to reproduce the very large number of microphotographs which would have been necessary to make the report fully intelligible. But we considered that it was not our duty to provide original memoirs for the reader, and that it would suffice for us to give a sufficiently complete idea, so that anyone could get, from our report, an exact idea of the works which have been analysed, and then resort to the authors in case of need.

I.

Progress of Technical Metallography.

A. Preparation of Polished Surfaces. Of the three operations, cutting, grinding and polishing, required for preparing a polished section, the latter, which used to be the longest and most laborious, has been largely improved by Le Chatelier[1]). Thanks to him, the rouge which was formerly used, has been replaced in most cases by chromium oxide, and especially by alumina: the latter is obtained by the calcination of ammonium alum and is washed by Schlœsing's method, as used in the mechanical analysis of the kaolins. Alumina thus prepared gives the same result as rouge, with great economy of time and labour.

[1]) Bulletin de la Societé d'Encouragement pour l'Industrie nationale, 5, VI, 365, September 1900.

The surfaces thus obtained, perfect in appearance and doubtless quite sufficient for determining the structural constituents, do not, however permit of certain difficult investigations, which we shall have the honour of submitting to the Congress in a personal communication. Whatever may be the method of polishing, the superficial film has been changed by the grinding; according to Beilby's[1]) observation, the polishing smooths hardened surface without gettig rid of it. It is however necessary to get rid of it, if one deares to study the intimate nature of the mechanical deformations.

Success can be obtained by using chemical reagents, properly chosen, which dissolve the metal whilst keeping the plane surface of the preparation, and after dissolution, the surface is repolished with alumina on a damp cloth. Prof. Ischewsky[2]) working in Le Chatelier's laboratory, has found a reagent for iron and steel, consisting of a solution of picric acid in absolute alcohol, which is very successful.

B. Reagents. The Ischewsky reagent is excellent for the purpose of differentiating the constituents of steel, and is thus of general interest on account of the preponderance of iron in metallurgy. Speller[3]) recommends a solution of 2 to 4 cm^3 of ordinary nitric acid, in 100 cm^3 of glycerine. The reagent can be applied with the end of the finger on the polished section; the ferrite remains brilliant, even after a continuous application of 24 hours duration.

C. Preservation of the Preparations. Le Chatelier[4]) uses Zapon varnish, a solution of gun-cotton in amyl acetate. A. Richards[5]) recommends a solution of paraffin in benzine.

D. Photography. Le Chatelier has also made marked progress by photography, notably by using monochromatic light obtained from the mercury vacuum lamp and interposing suitably

[1]) Proc. Roy. Soc., LXXII, 226; also Revue de Métallurgie, 1, abstracts, 33, January 1904.

[2]) Stahl und Eisen, XXIII, 120, 15. January 1903.

[3]) Métallographist, VI, 264, July 1903.

[4]) Loco citato.

[5]) Metallographist, VI, January 1903.

coloured screens. These coloured screens absorb a great deal of light. They can only be used with a strong source of light, without running the risk of over-exposure. We are quite satisfied with the Nernst lamp. Most of the American laboratories use the ordinary arc-lamp with automatic regulator.

II.

Equilibrium curves.

We will now inspect, in order of increasing complication, the groups of alloys which have been examined by means of metallography since the middle of 1901.

A. Gold and silver. On certain properties of the alloys of the gold-silver series; Sir W. C. Roberts-Austen and T. Kirke Rose, Proc. Roy. Soc., LXXI, 161. Metallographist, VI, 155, April 1903.

The freezing-point curve (fig. 1), determined by automatic pyrometer, is at first almost horizontal, starting from the melting-point of gold, then descending and finally joining the melting-point of silver. The absence of any critical point proves that all the alloys of this series are isomorphous mixtures, solid solutions, as announced by Gautier in 1896. Even an annealing, lasting for two months, at a constant temperature of 700° C. (during the day) and falling to about 100° C. during the night, has not revealed the presence of any eutectic.

B. Silver and copper. Osmond. Imperfect equilibria in alloys. Iron and steel Metallurgist and Metallographist, 1904.

The silver-copper series is the well-known type of two metals which do not form definite compounds. It has already been taken as an example by one of us at the conference held in 1897, at the Stockholm Congress, before the »Association Internationale«. The study has however been completed more recently.

The curve ACB (fig. 2) is the freezing-point curve or the »liquidus«, as Prof. Bakhuis Roozeboom calls it. All the alloys form a single liquid homogeneous phase above ACB. According to percentage, silver begins to solidify in accordance with curve AC, copper following BC, and the solidification takes place at

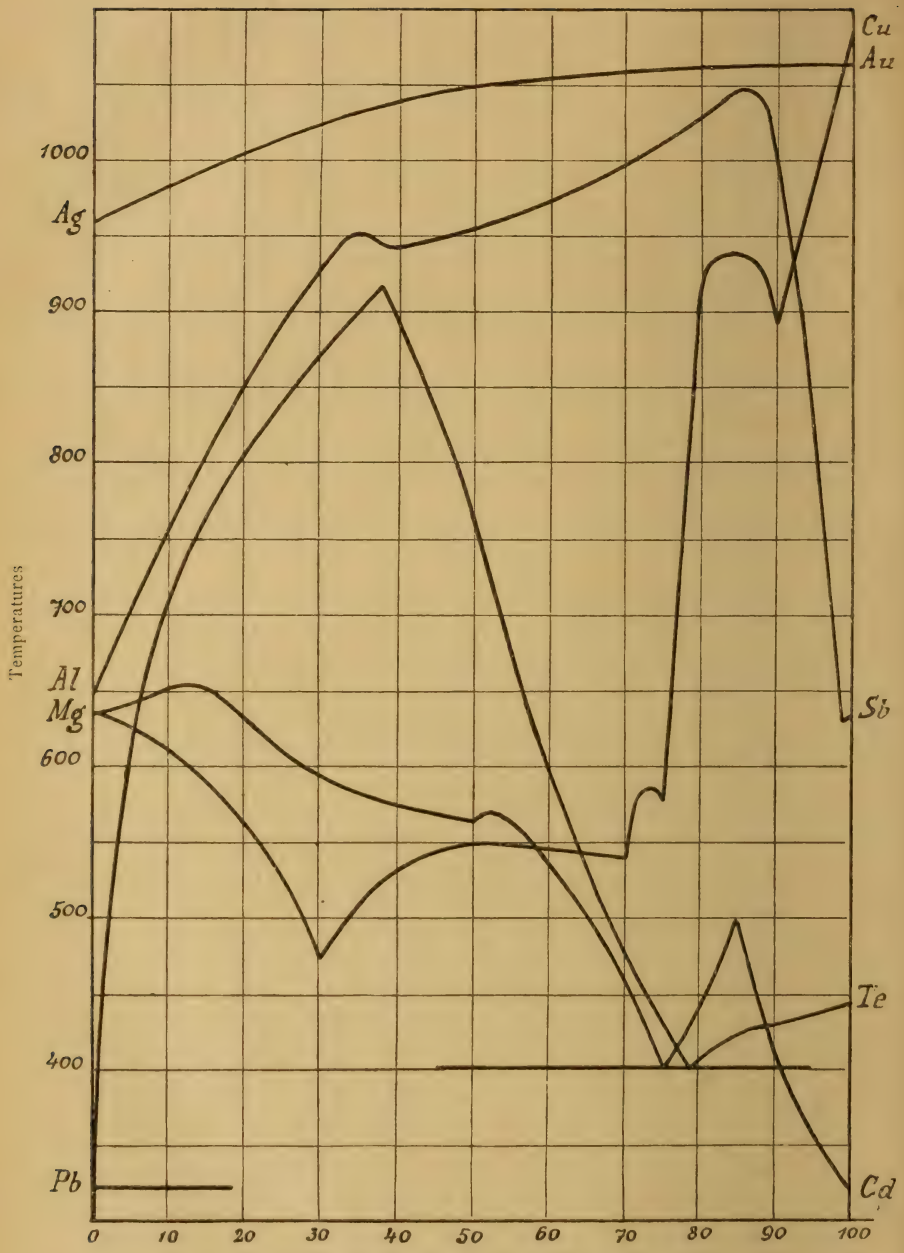

Cu
Au
1000
Ag
900
800
700
Al
Mg
600
500
400
Pb
Sb
Te
Cd
Temperatures
0
10
20
30
40
50
60
70
80
90
100
Composition in percent by weight.

Fig. 1.

diminishing temperatures. If the solubility of the two metals were nil at the solid state, the solidification would in all cases terminate at constant temperature, indicated by the horizontal line through C in the presence of a copper-silver eutectic, and the eutectic line would extend from axis AO to axis BP.

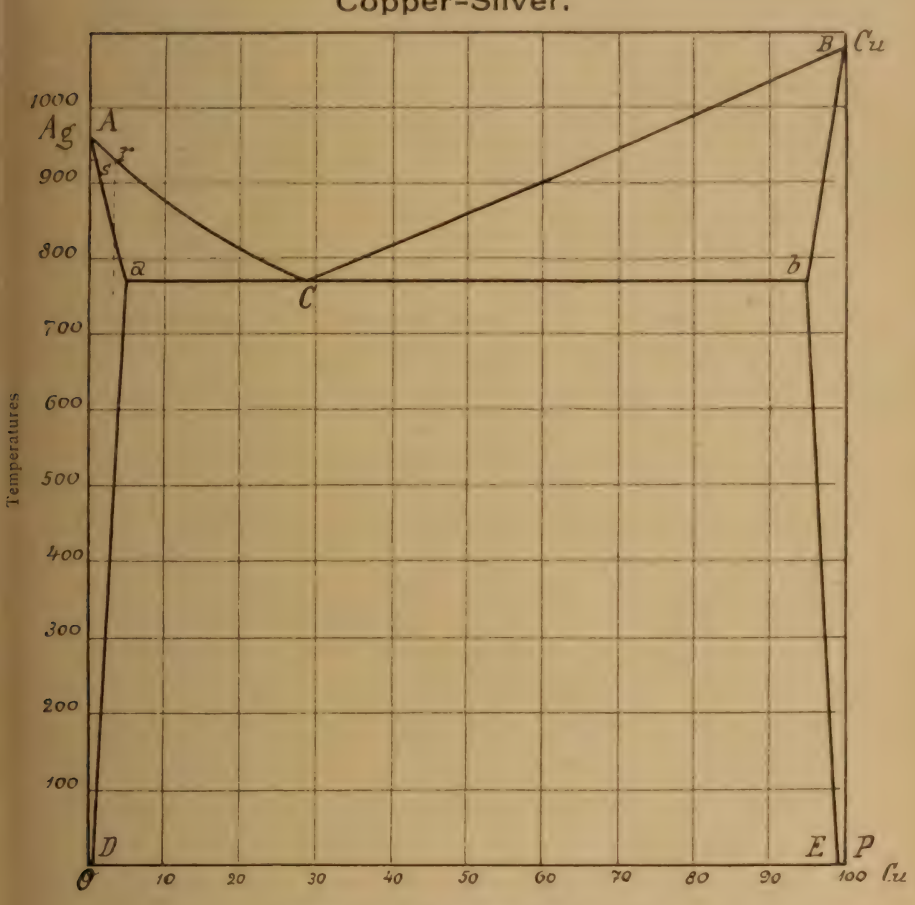


Fig. 2.

The solid silver is however a little soluble in the solid copper, and vice versa. The result is, that the eutectic line passing through C, stops at points a and b, whose positions have not been exactly determined, but it is certain that they are at appreciable distances from the axes.

Taken together the lines Aa, aCb and Bb form the complete curve of solidification, the »solidus« as Roozeboom calls it. Thus the alloys which are lower in copper than a, or higher than b, are not eutectiferous. Let us consider an alloy whose copper percentage is given by the abscissa to point r; it will begin to solidify at a temperature represented by the ordinate to r. According to a law discovered by Roozeboom and verified by experiments, it will deposit a solid whose composition is given by s, the point of intersection between the horizontal through r and the „solidus" Aa; the liquid then becomes richer in copper and deposits a new solid equally rich in copper. But if the cooling is sufficiently slow, the copper will distribute itself uniformly in the deposit, and the solidification will finally end at the junction of Aa with the vertical through r; giving a solid homogeneous solution of the initial composition.

It is only for the alloys which are higher in copper than at a, or lower than at b, that the process of solidification, the same as in the beginning, leads to the production of an eutectic, when the progressive concentration of copper for instance has led to the percentage at point C.

The two phases which constitute the eutectic are naturally saturated the one with the other at the temperature of aCb, but the solubility diminishes with the temperature; the silver saturated with copper, will deposit copper in accordance with aD; the copper saturated with silver will deposit silver in accordance with bE; the lines aD and bE indicate, for each solution, the mutual solubility at each temperature.

Such is the theory. But if the cooling is not sufficiently slow, as some time is required for establishing equilibrium, it may produce lag.

As the molecular mobility continues to diminish when the temperature falls, and can become nil at ordinary temperature, it may happen, and it does happen, that the retarded equilibria are never established, nor even, do they begin to be established in the temperature-zone where the molecular migrations have become impossible.

We have two causes producing heterogeneity in the cooled alloys:

1. The copper, for instance, has not been able to distribute itself uniformly in the crystallites of first consolidation, which have kept it in solution.

2. The copper, for instance, which has been separated from the solid solution in accordance with aD, has not been able to rejoin the copper isolated in the eutectic, in the case of alloys which contain an eutectic, or has not been able to individualize itself in the others.

From this arises a progressively variable composition of crystallites starting from their axes. Thus, in a silver alloy containing one per cent of copper and which has not been cooled very slowly, copper is not found in discernible grains or even traces. At ordinary temperature, the solid silver cannot keep this proportion of copper in solution. Copper is certainly separated but has not been able to reassemble, and when oxidising the sample, one finds only a progressive concentration of copper towards the points where the eutectic is liquated in the alloys which contain one, that is to say towards the regions of the last consolidation. This concentration is shown by a graded brown halo. This is a very general phenomenon.

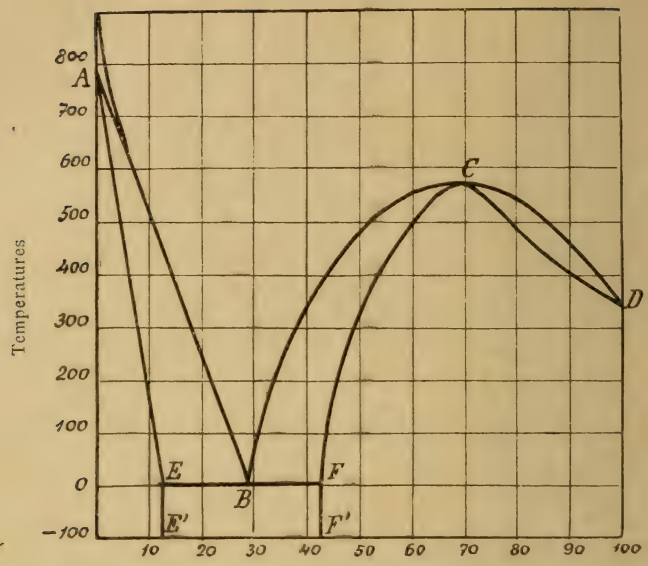
C. Iron and nickel. Osmond and Cartaud: sur les fers météoriques; Revue de métallurgie, I, Mémoires, 69, February 1904.

Fig. 3 is the diagram of equilibrium of the ferro-nickel series, during cooling. This diagram is only partial; it does not contain the freezing-point curves which are unknown, but only the curves of allotropic transformation in the solid state. Freezing-point curves and transformation curves are governed by the same general laws; the latter only are more subject to petardations.

We will neglect the small part of the diagram in which the two known transformations of iron are still distinct.

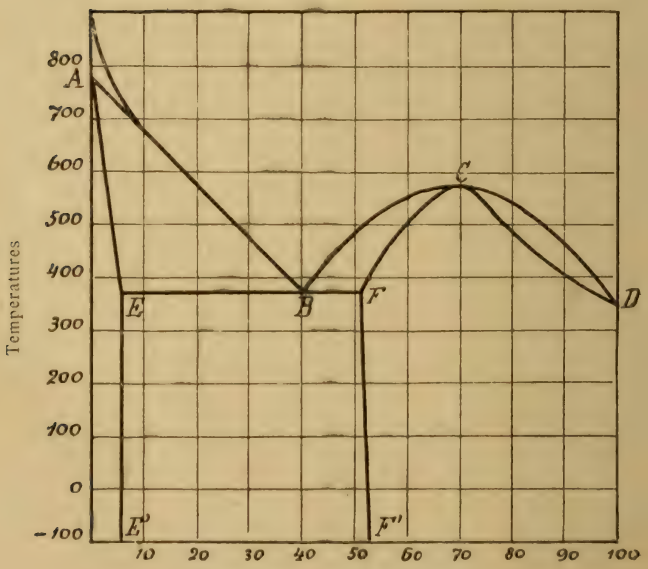
Admitting this simplification, the lines $ABCD$ represent the beginning of the transformations; they have been determined with sufficient exactness from tests made by Hopkinson, Le Chatelier, Guillaume, Dumont, Dumas and by one of us. The curves AE and FCD of the final transformation are only known in first approximation, the lines EF, EE' and FF' are purely hypothetical.

Iron-Nickel.



Nickel in percent by weight.

Fig. 3a.



Nickel in percent by weight.

Fig. 3b.

The part of the diagram which has not been determined by test, was drawn as early as October 1900 by Prof. Bakhuis Roozeboom and sent to us in a private communication, as the most probable result of facts known at that time by comparison with similar cases; this under all reserve.

It will be seen that while the percentage of nickel increases from 0 to 29 (these figures have been determined from Guillets last researches), the transformations fall off until near 0° C. As the percentage of nickel still increases, the curve rises rapidly, passes through a maximum point C' about 580° C. and 67 per cent of nickel, and then descends to join the point of transformation of pure nickel.

In similar curves, the presence of a maximum often indicates a definite compound; but this is a particular case of a more general fact, the identity of composition of the phases above and below the maximum. In the example just considered, there is no appearance of a definite compound.

Above $ABC'D$ there is in all probability a homogeneous solid solution of γ iron and of β nickel. Below AE there is a solid solution of α nickel in the α iron, and below FCD a solid solution of α iron in the α nickel. Within the region of $EFE'F'$ there should be a complex of two conjugated solutions containing OE and OF per cent of nickel respectively. In fact the micrographic analysis only reveals solutions wich are incompletely transformed or separated, and forms of transition between the different allotropic states of iron.

In these alloys the eutectic line EF, or to use a new expression of Prof. Howe's, who distinguishes between solid eutectics and liquid eutectics, the eutectoid EF is only virtual. The transformations begin at a temperature too low to be finished, and all the alloys between E and F are metastable.

The meteoric irons which are, as is well known, alloys of iron and nickel, justify however the diagram completely. The two conjugated solutions predicted by theory are actually found in them, and have been known for a long time by the names of kamacite and taenite. The kamacite contains always from 6 to 7 per cent of nickel, and the taenite contains more than 35 per cent of nickel.

Why then are the theoretical predictions verified only by the super-terrestrial alloys? The reason seems very simple. In these alloys of nickel and iron, the transformations during cooling undergo enormous retardations, which is shown by the difference between the respective positions of the line *AE* in the cooling diagram (fig. 3a) and in the heating diagram (fig. 3b). For this reason, the alloys whose percentage of nickel is less than *OB* are said to be irreversible. It is probable that the cooling and heating curves would coincide and approach more nearly the heating curves, if the cooling were infinitely slow, and this infinitely slow cooling is found realized in the meteorites, which are fragments of broken up planets. The transformations which are only partially accomplished in the terrestrial alloys at nearly ordinary temperature, have been accomplished, and the phases separate between 350° and 400° C.

D. Iron and copper. J. E. Stead, Notes on alloys of copper and iron. Journ. Iron and Steel Inst., 1901, II, 104. Metallographist, V, 25, January 1902.

The previous data relative to these alloys were already very numerous, but also very contradictory. By a methodical study and especially by a judicious application of metallography, Stead has thrown much light on this question.

Liquid copper and iron alloy in all proportions. The solid alloys divide themselves into three classes:

1. The alloys, containing between 0 and 2·73 per cent of iron, slightly magnetic, are formed of one single phase, a homogeneous solid solution.

2. The alloys containing copper up to 8 per cent, are also composed of a single phase. However the homogeneity is not perfect: on heat-tinting the polished section there appears a cellular network with graded boundary lines, the centre of the cells is richer in iron than the surface. The result of this is that equilibrium was not attained in the tested specimens. This is a similar case to that of the copper-silver alloys which we mentioned above.

3. The alloys containing between 2·73 and 92 per cent of iron, contain crystallites formed of iron which is saturated with copper and surrounded by a second constituent which seems to

be an eutectic, but this has not yet been decided. This second constituent is more fragile than the crystallites; it is across its mass that the fractures are propagated.

These results seem to bring the iron-copper series nearer to the iron-nickel series, but they are not yet sufficiently complete to allow the diagram of equilibrium to be plotted.

If iron be replaced by steel or cast iron, the two metals, according to early observations of Mushet, do not alloy any more whatever the proportions in the liquid state. They form two conjugated alloys which lie above each other according to their densities. The proportion of copper which can alloy with iron is limited to about 7·5 per cent in a gray hematite iron of 3·4 of carbon and 3 of silicon; it can reach 15 per cent in a steel of one per cent carbon, but an important part of the copper is isolated in globules in the cooled metal, and the solid solution retains probably not more than 7 per cent.

An alloy of equal parts of iron and copper, re-cast in the presence of carbon, takes up carbon and separates into two layers, one containing 2 per cent of carbon and 10 of copper, and the other 10 per cent of iron and 0·08 of carbon.

The copper seems neither to retain carbon nor to bring on the separation of graphite.

The formation of pearlite is retarded.

The copper is not dangerous for casting; it increases the tenacity of foundry iron.

E. Lead and tellurium. H. Fay and C. B. Gillson. The alloys of lead and tellurium. Trans: Amer. Inst. Min. Eng., XXXI. 527, 1901.

Fay and Gillson have made researches for the purpose of finding a use for waste tellurium.

The freezing-point curve (fig. 1) shows a maximum at 917° C. corresponding to the definite compound *PbTe*, and a minimum at 400° C. with an eutectic line, which is the place of the simultaneous solidification of tellurium and of lead-telluride.

Micrography confirms the indications of the curve. The alloys of antimony and tellurium are rather similar to the preceding ones, and give a definite compound Sb_2Te_3. But this compound is here isomorphous with the antimony, while it forms an eutectic with tellurium.

F. Aluminium and other metals. Guillet, Contribution à l'étude des alliages d'aluminium, Bull. Soc. d'Encouragement, (5), 1, CIII, 221, August 1902.

The freezing-point curve of the alloys of aluminium and antimony has been determined by Gauthier[1]) and is reproduced in fig. 1. It shows two maxima, the one corresponding to the definite compound $SbAl$, the other is close to $SbAl^{10}$.

Guillet believes that it has confirmed the existence of these two compounds.

He has likewise studied the alloys of aluminium with many other metals and describes a large number of new compounds. We will not dwell too long on this otherwise important work, because it is above all things a work of classical chemistry. The microscope has helped as an accessory, and afterwards has been used to verify the results of separations and chemical analysis. The whole is an example of how to make use of the mutual help which Chemistry and Metallography can and ought to give to each other.

G. Magnesium and cadmium, magnesium and copper. Boudouard, Bull. Soc. d'Encouragement, CIII., 216, August 1902 and CV, 201, August 1903.

The freezing-point curve (fig. 1) of the alloys of the copper-cadmium series shows three maxima. Two of them show evidence of the existence of two definite compounds $CdMg$ and $CdMg^4$, the third one corresponds to the composition $CdMg^{30}$, but the analysis has not permitted of isolating a definite compound of this formula, which is besides rather improbable; it is more probable that $CdMg^4$ form solid solutions with magnesium, and that the solid solution of the composition $CdMg^{30}$ is in equilibrium with the liquid solution of the same composition. This is what we have already found in the transformation of the ferro-nickel alloys.

[1]) Bull. Soc. d'Encouragement, (5), 1, 1312, October 1896.

The curve of the alloys Magnesium-Copper (fig. 1) also shows three maxima. In this case, the three definite compounds, $CuMg_2$, $CuMg$ and Cu_2Mg have been isolated by appropriate chemical reagents. For micrographic examination, Boudouard has successfully employed the method of stratification of molten metals. The molten magnesium was poured into a crucible containing copper smelted under a layer of sodium chloride. The solidified bottom presented the whole scale of alloys on one single specimen.

H. Iron and sulphur. Le Chatelier and Ziegler, Bull. Soc. d'Encouragement, CIII, 368, September ARS 1902. Metallographist, VI, 19, January 1903.

The harmful influence which small quantities of sulphur exercises on iron and the remarkable action of manganese in counteracting this influence, have caused Le Chatelier and Ziegler to study more closely than has been done before, the relations of this non-metal with iron and manganese.

It is probable, a priori, that the sulphur in the cast irons and steels would be found in the same state as we know it in commercial sulphide.

The composition of this sulphide differs somewhat from FeS: the micrographic examination shows that it is formed of three constituents:

1. Free iron of white colour;
2. A yellow body, which is the definite sulphide FeS;
3. An eutectic of which one constituent is FeS and the other, which deposits copper from its solutions, is probably itself an eutectic, unresolvable by microscope, of metallic iron and the oxide FeO.

The sulphide FeS is at least dimorphous. It undergoes during heating and cooling, a first transformation which is reversible, which a knowledge of dilatation easily makes evident, and which electrical resistance shows ought to be placed in the vicinity of 140^0 C. This change of state is analogous to that which causes the disintegration of heated litharge. The dilatation seems to indicate a second one a little above 220^0 C.

The commercial ferrous sulphide melts at about 950^0 C., giving off gas; the pyrite FeS_2, when heated, loses sulphur; if the heating is extended to 1500^0 C., the percentage of sulphur

is only half of that which corresponds to the formula FeS; the microscope indicates that there is then a mixture of FeS and of Fe, the latter being the element of the first consolidation. The eutectic has disappeared.

In fact when treating combinations of iron and sulphur in an oxidising atmosphere, and when the atmosphere remains oxidising, one has really to do with the ternary mixture Fe, FeS and FeO; just as in the industrial proceedings of refining, or in the preparation of sulphide in laboratories.

The following reaction between these three substances may be considered:

1. Dissociation of ferrous sulphide into sulphur and iron:

$$2FeS = 2Fe + S_2$$

2. Ferrous oxide reacting on the sulphide and producing a «spitting»:

$$2FeO + FeS = 3Fe + SO_2$$

3. Equilibria of saturation are established between the metal, the sulphur and the oxide at the contact of the molten mixture:

These multiple reactions, skilfully discussed and explained account for the observed complex facts.

In the solid irons, the solubility of sulphur seems to be almost negligeable, and the authors have not been able to find any certain indication of a solid solution, as for instance is the case in the irons containing phosphorus. In the low percentages, the sulphide, mixed a little with its eutectic, forms round specks, chiefly at the junctions of the grains; as the percentage increases, the areas become oval, then become more and more elongated, tending to unite into a network which is visible in ordinary polishing. Etching by picric acid shows that the iron corrodes more quickly at its junction with the sulphide, and, when the sulphide is scattered in grains, it appears as a special network, binding these grains. The supposition of an appreciable solid sulubility, at any rate at high temperatures, is not quite impossible.

One can easily conceive that the ferrous sulphide, a substance of very little tenacity when cold, and fusible at about 950° C., diminishes considerably the strength of iron when cold, and destroys it completely when hot.

The influence of manganese is explained with the same facility. In the alloys of manganese and sulphur, the sulphide MnS of green colour is separated in crystallites produced in the first consolidation, whose fusing-point is higher than that of manganese and, consequently, of iron. Moreover it is only slightly soluble in manganese.

In the liquid irons and steels, the manganese will carry with it all the sulphur, and by liquation isolate the greater part of it. This is exactly what does take place in practice. The other part of the sulphide which remains imprisoned in the metal during cooling is found scattered in a state of small isolated grains which cannot do any appreciable harm.

In fact the sulphur is divided between the iron and the manganese, but the work was done with small essay-ingots which cool rapidly. In ingots as used in practice and which always contain, unless by accident, more manganese than is required to saturate the sulphur, our authorities have never met with FeS.

These researches by Le Chatelier and Ziegler, as important for their, practical consequences as for a model of scientific discussion render almost needless what has been previously published by Professors Arnold and Waterhouse [1]) on the same subject.

I. Iron and silicon. T. Baker: The influence of silicon, on iron, Journ. Iron and Steel Inst., 1903, II, 312; Guillet: Aciers au silicium; Revue de métallurgie, I, mémoirs, 46, January 1904.

The relation between iron and silicon has been until now chiefly studied by chemists, especially by Lebeau [2]), who has isolated the three definite compounds Fe_2Si, $FeSi$ and $FeSi_2$. But Fe_2Si, the poorest in silicon, appears only as a constituent of alloys above a somewhat high percentage, certainly above 10 per cent in the absence of carbon. In lower percentages, silicon is present in solid solution, but it is not known in which form. Baker, and also Guillet, have entered upon this question by the aid of micrography, and have produced similar results without however having succeeded in answering it.

K. Copper and antimony. Baykoff: Bull. Soc d'Encouragement, CIV, 626, May 1903.

[1]) The influence of sulphur and Manganese on Steel, Journ. Iron and Steel Inst., 1903, part 1, 136.

[2]) Comptes rendus de l'Académ. des Sciences, CXXXII, 681, 18 March 1901.

The alloys of copper and antimony have already been the object of much research-work. Le Chatelier has determined the curve of first solidification, which showed the existence of a definite compound[1]) and the co-efficients of dilatation[2]). The study of the microstructure has been entered upon by Charpy[3]) and pushed still further by Stead[4]), who has also characterised the two definite compounds $SbCu_2$ and $SbCu_3$. But the problem was more complicated than had been expected. There still remains, something to be done.

Professor Baykoff has taken great trouble in re-plotting the heat curves, but this time extending them beyond the solidification, and with great skill he has interpreted them by means of metallography.

The curve of first solidification is formed of 5 branches (fig. 4) each corresponding to an isolation of the following crystals:

A B. Antimony;

B C. The compound $SbCu_2$;

C D. A solid solution of antimony in the definite compound $SbCu_3$;

D E. A solid solution of copper in the same compound $SbCu_3$;

E F. Copper probably containing a little antimony in solution.

The maximum *D* (61 per cent of copper) corresponds to $SbCu_3$, fusible at 681° C. It should be noticed that $SbCu_2$, the well known violet compound, does not cause a real analogous maximum. This is because it melts on being decomposed at 587° C. (line *C H*). The reaction, analogous to the fusion of a salt in its crystallisation water, for instance:

$$SO_4Na_2,\ 10\ H_2O \rightleftarrows SO_4Na_2 + 10\ H_2O$$

is here

$$2\ SbCu_3 + Sb \rightleftarrows 3\ SbCu_2$$

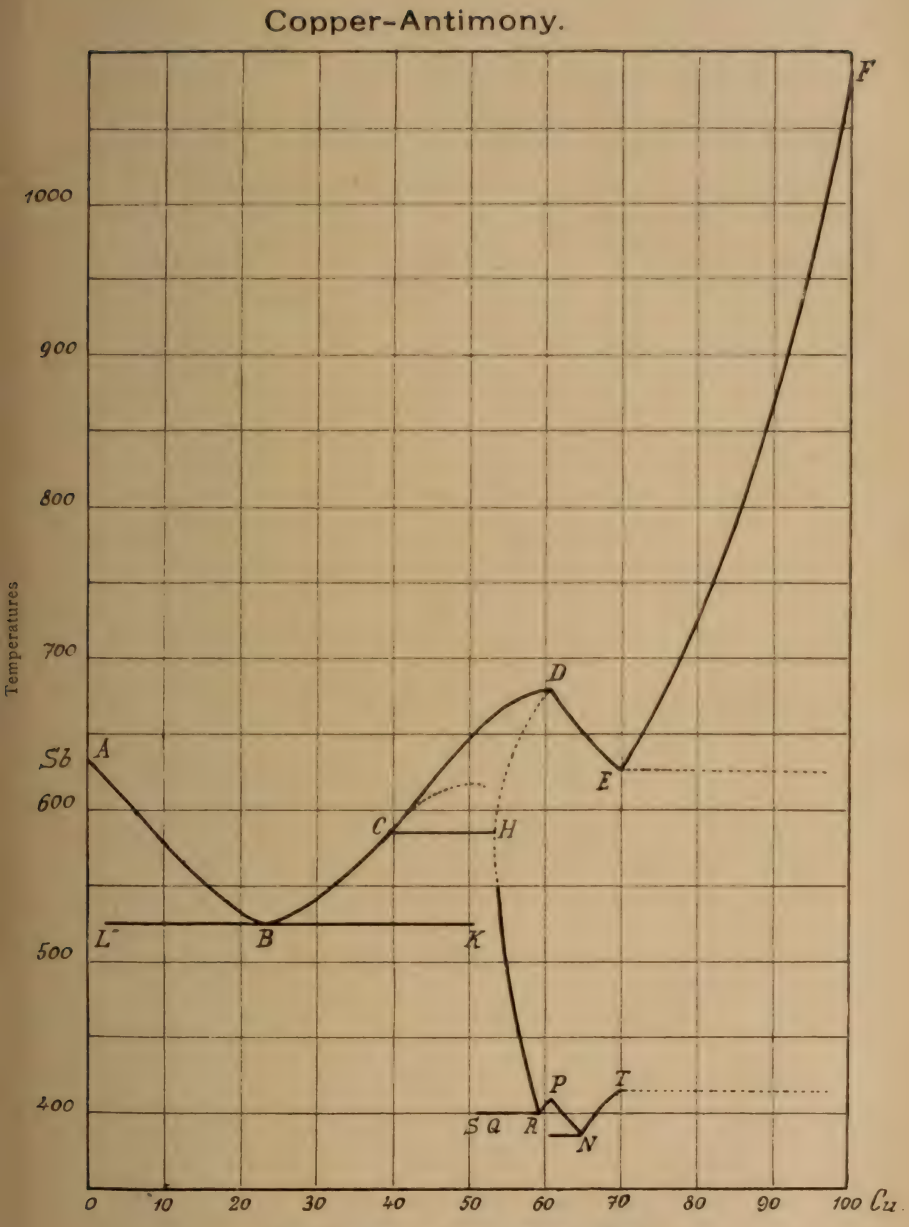
Finally all the alloys between *O* and 50 per cent copper are after cooling, composed of crystals of Antimony or crystals of $SbCu_2$, cemented by the same eutectic $Sb + SbCu_2$, although, between *C* and *D*, the first deposit is a solid solution of Antimony in $SbCu_3$.

[1]) Bull. Soc. d'Encouragement, (4), X, 574, May 1895.

[2]) Comptes rendus Ac. des Sciences, CXXVIII, 1444, 12 June 1899.

[3]) Bull. Soc. d'Encouragement, (5), II, 397, March 1897.

[4]) Journ. Soc. chemical Industry, XVII, 31. December 1898.



Composition in percent by weight.

Fig. 4.

When the percentage of copper goes beyond 51 ($SbCu_2$), the same reaction

$$2\,SbCu_3 + Sb \rightleftarrows 3\,SbCu_2$$

is produced again, following the branch HR, but at a rapidly falling temperature; because of the absence of antimony, the reaction ceases to act on the whole of $SbCu_3$ and is now equivalent to the separation of $SbCu_2$ from an excess of $SbCu_3$.

On the other hand, $SbCu_3$ is dimorphous and is transformed at 407° C (point P) when it is pure, following the curves PR or PN in the alloys which contain an excess of antimony or of copper. The curves HR and PR intersect at a point R which is thus an eutectoid point, with its eutectoid line SQR for the alloys between $SbCu_2$ and $SbCu_3$ (51 and 61 per cent of copper). Finally the branch TN corresponds to the separation of copper from its solid solution in $SbCu_3$ and produces a new eutectoid point N.

The existence of these critical curves below the solidus, makes one expect, in the region considered, a difference of structure between the quenched alloys and the same alloys slowly cooled. In the alloys between 53·5 and 69 per cent of copper, after quenching from a temperature taken between $HRPNT$ and HDE one only finds one single homogeneous solid phase, to which are joined the copper crystallites in the alloys richer in copper. The corresponding alloys slowly cooled are, on the contrary, complexes, except of course pure $SbCu_3$.

The quenching causes the suppression of two transformations, the separation of $SbCu_2$ from the solid solution of Antimony in $SbCu_3$ and the dimorphism of $SbCu_3$.

When, on the contrary, equilibrium has been established by means of slow cooling, the structure gives evidence of the two phenomena. In order to give to each of them its due share of importance, Prof. Baykoff makes use of two kinds of etching on the same alloy; one of them must be strong to show that the alloy is not homogeneous; the other must be weak, to manifest the change of the characteristic structure of polymorphism. For the alloys with excess of antimony, the best reagent is water. $SbCu_3$ is the first to undergo oxidation, while $SbCu_2$ remains unaltered.

If then etched by weak hydrochloric acid, the oxides are dissolved and the surface becomes white; but at the same time one perceives a fine and characteristic marking, which owes its origin to dimorphous transformation.

The alloys above 70 per cent copper require still further investigations.

L. Copper and tin. Heycock and Neville: on the constitution of the copper-tin series of alloys, Philos. Trans. Roy. Soc., (A), CCII, 1, 1903. (Bakerian Lecture.)

On account of their commercial importance the alloys of copper and tin have already given rise to numerous researches, of which many are important. The freezing-point curve has been plotted approximately by Le Chatelier[1]), and with many details by Heycock and Neville[2]). Roberts-Austen and Stansfield in the third and fourth reports to «Alloy Research Committee»[3]) have made a complete thermic study extending to phenomena produced after the first solidification. On the other hand, Guillemin[4]), Behrens[5]), Le Chatelier[6]), Charpy[7]), Stead[8]) and Campbell[9]) have entered upon micrographic analysis, and have pushed their investigations more or less far.

Profs. Heycock and Neville have proposed to complete the work of their predecessors, and to make a complete list of all the possible phases, and to assign to them their exact regions. Except for some points of detail still unsettled, they have fully succeeded therein by following the doctrines of Roozeboom, and applying the methods which have already proved successful in examining the alloys of the ferro-carbon series.

[1]) Bull. Soc. d'Encouragement, (4), X, 574, March 1895.

[2]) Phil. Trans. Roy. Soc., (A), CLXXXIX, 25, 1897.

[3]) Proc. Inst. Mech. Engs., April 1895 and February 1897.

[4]) Rapport à la Commission des méthodes d'essai des matériaux de construction, April 5. 1893; 1er session, tome II, 19, Paris, 1895. — Baumaterialienkunde, Heft 4, 1897—1898.

[5]) Mikroskopische Gefüge der Metalle und Legierungen, Voss, Leipzig, 1894.

[6]) Bull. Soc. d'Encouragement, (5), I, 559, April inrt. 1896.

[7]) Bull. Soc. d'Encouragement, (5), II, 384, March 1897.

[8]) Journ. Soc. chemical Industry, 30 June 1897.

[9]) Paper read on December 20, 1901, before the Inst. of Mech. Engs.

The alloys slowly cooled down to ordinary temperature preserve the trace of the various equilibria successively gone through. As Heycock & Neville say, the work which consists of explaining the structure, recalls that of paleographists obliged to decipher on one palimpsest manuscript writings of different ages. It was therefore necessary by chilling, starting from temperatures conveniently chosen between the critical points, to stereotype as much as possible the successive structures corresponding to the passages through each region. This in fact was the best method.

The results are condensed in diagram fig. 5.

The curve $ABLCDEFGHIK$ is the liquidus.

The sharp points C, D, G and I indicate as many abrupt changes in the nature of the solid phase, which begins to be deposited in equilibrium with the liquid.

The solidus is the broken line $Ablcmdef E_2 E_3 H' H'' K'$. The exact shape of Ab and the positions of m and H still leave some uncertainty.

The area between the liquidus and the solidus represents the mixtures of liquid and solid.

The recognised solid phases are seven in number, and are denoted by the letters α, β, γ, δ, b and H, the seventh being pure tin or nearly pure tin.

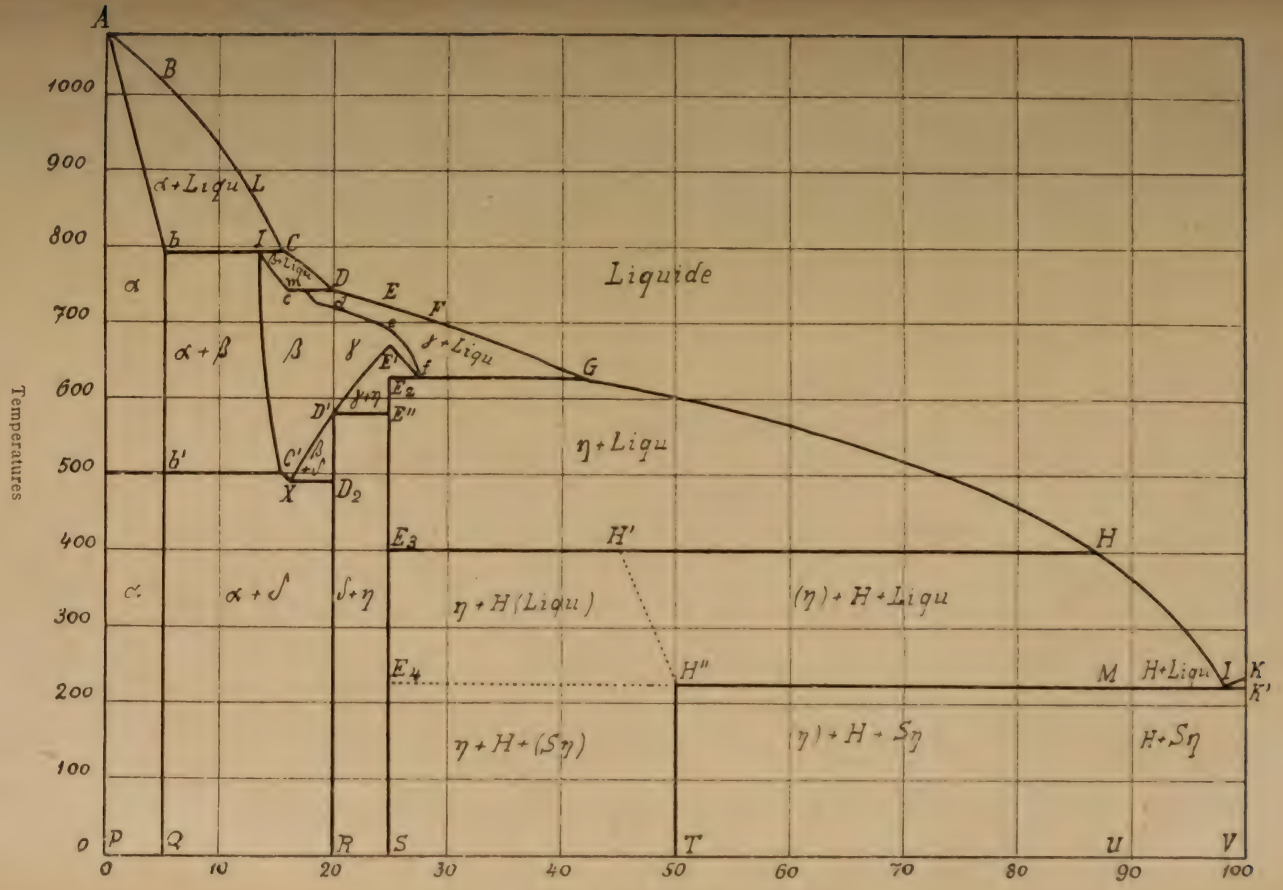
The crystals α, an isomorphous solid solution with copper, can contain from 0 to 9 per cent of tin by weight.

The crystals β are another solid solution, perhaps isomorphous with α, their percentage in tin (by weight) is from 22·5 to about 27.

The crystals γ are also a solid solution of the two metals or their compounds; they do not appear to be isomorphous with the preceding ones, and contain from about 28 to 57 per cent of tin by weight.

δ is perhaps still a solid solution but is more probably the definite compound Cu_4Sn.

η is the definite compound Cu_3Sn between E and H, and continues between E and D with the same crystalline forms, but perhaps becoming a solid solution.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L
b
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C
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D
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G
α+β
β
γ
E'
f
γ+η
E2
D'
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b'
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(η)+H+Liqu
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100
0
10
20
30
40
50
60
70
80
90
100
Temperatures
Composition in atomic percent of tin.

Fig. 5.

H seems to be the compound Cu_3Sn containing a little Cu_3Sn in solution.

α, β, γ, η and H are primary crystals which are separated directly from the liquid; δ results from the recrystallisation of the unstable solid solutions β and γ.

At point C is produced the isothermal transformation

$$\alpha_b + \text{liq}_c = \beta_1$$

and at point D the transformation

$$\beta + \text{liq} = \gamma$$

or perhaps simply

$$\beta = \gamma$$

in which case the points c and m will coincide.

The branch $ABLG$ deposits the mixed crystals α
" " CD " " " " β
" " $DEFG$ " " " " γ
" " GH " Cu_3Sn (η)
" " HI " H ($CuSn$ with a little of η)
" " IK " Sn

The alloys are grouped as follows:

Alloys AB (from 0 to 5 atomic per cents of tin). The theory predicts a single homogeneous phase α. In fact the photographs show the crystallites α, cemented by a residue of mother-liquor. But this mother-liquor is completely absorbed by the crystallites when annealing at a temperature a little lower than bC'.

Alloys BL (of 5 to 13·5 atomic per cents of tin). Above bC, they are constituted of a single phase α. In bC they are dissociated in a mixture of crystals α, compositions b, β and 1. In the region $bb'C'$ the crystals α grow at the expense of the crystals β, which play the part of mother-water. At the eutectic temperature $b'C'$ (500° C.), the crystals α are broken up in a complex $\alpha + \delta$.

Alloys LC (of 13·5 to 15·5 atomic per cents of tin). At the temperature bC, the crystals α of first consolidation are changed into β, which form, below the solidus, the whole of the alloy. In cooling this solution β deposits α, and the surplus is divided into $\alpha + \delta$ at the temperature $b'C'$, as with the alloys BL.

Alloys CD (from 15·5 to 20 atomic per cents of tin). The alloys from 15·5 to 16·5 solidify directly to the state of crystals β. From 16·5 to 20 Sn, it forms also first crystals β but which do not fill up the total volume, and which appear to be transformed temporarily below temperature D into crystals γ. Between cd and $C'XD'$, the solution appears to be homogeneous. Following $C'XD'$ it deposits δ, and the surplus is transformed into $\alpha + \delta$ when the temperature reaches XD_2. The abnormal fact that the eutectic point C' of the alloys LC' is a little higher than the eutectic point X of the alloys CD, may be explained for the latter, by a slight retardation due to the absence of germs α. It is possible that the phase δ is the compound $SnCu_4$. Although this alloy of composition $SnCu_4$ undergoes during cooling an exothermic transformation at D', it remains practically the same after this transformation; there are however indications of re-crystallisation at point D'. It is possible that the compound $SnCu_4$ does not exist above D'.

Alloys DE (from 20 to 25 atomic per cents of tin). The homogeneous crystals γ deposit along $E'D'$ the lamellar crystals γ_1, which are the compound $SnCu_3$ pure or containing $SnCu_4$ in solution. Below $D'E'$ we have a complex $\gamma + \gamma_1$ and the line $D'E''$ corresponds to the transformation of γ into δ.

Alloys EF (from 25 to 27·5 atomic per cents of tin). Behave at first as the preceding ones, and give successively $\gamma +$ liquid, γ solid, and $\gamma + \gamma_1$. But at the temperature of fG, the residue of γ breaks up into $\gamma_1 +$ liquid of the composition G. This partial fusion of a solid in the course of cooling is a phenomenon which deserves special notice.

It may be noted that the triangular region Ixf is that of solid solutions which break up into two phases by cooling, and nothing in the cooled alloy could make one suspect their existence.

Alloy FG (from 27·5 to 42 atomic per cents of tin). The crystals γ of first consolidation, when they reach, with the composition f, the temperature fG, break up into $SnCu_3 +$ liquid. Below fG, the liquid deposits new crystals of $SnCu_3$ and becomes richer in tin, until the temperature has fallen to 400° C. Starting from 400° C, the same takes place as in the following group.

Alloys GH (from 42 to 87·5 atomic per cents of tin). The liquid deposits $SnCu_3$, until it reaches the composition H and the temperature 400° C. At this temperature γ_1 reacts on the liquid, to

produce the phase H ($SnCu$ containing a little dissolved $SnCu_3$). The reaction is very slow, the crystals of H deposited on η_1, retard it more and more, and the equilibria are very difficult to establish. Hence the frequent presence in certain regions of a third abnormal phase denoted in the diagram between parentheses.

Alloys HI (from 87·5 to 98·3 atomic per cents of tin). The first solid formed is H.

Alloys IK (from 98·3 to 100 atomic per cents of tin). The tin is deposited first and surrounded by an eutectic $Sn + H$.

M. Iron and Carbon. Utilising the thermic researches of Roberts-Austen[1]), and guided, in order to explain them, by his grand experiment, Prof. Bakhuis Roozeboom has drawn the diagram of equilibrium (fig. 6) for the alloys of the ferro-carbon series. This diagram cannot yet be considered as absolutely final in all its parts for want of sufficient experimental data. But it can already serve as base for the teaching of scientific metallurgy and has caused several recent researches, having for their object either the exploration of new regions, or the solution of difficulties held in suspense.

Thus Prof. Stansfield[2]) has tried to find a criterion for specifying the meaning, still vague, of the terms »burnt and overheated«.

In diagram fig. 6 Prof. Roozeboom has introduced line Aa which has not been found by experiment, and which represents the end of the solidification of alloys lower in carbon than abscissa a. It is, in other words, a part of the solidus. A steel of carbon percentage tp heated to a temperature p within the region AaB (austenite + liquid) divides into a solid solution of carbon percentage ts and a liquid of percentage tr.

If quenched from point p, the liquid portion will furnish a structural constituent which can or cannot deposit the cementite, according to whether it is more or less rich in carbon, and which is up to a certain point new. According to unpublished observations of Osmond, this constituent possesses characters which make it approach micrographically to troostite; but it is rather paradoxical to find troostite a constituent of a mild and incomplete quenching, there,

[1]) Fifth report to the Alloys Research Committee. Proc. Inst. Mech. Engs., Feb. 1899.

[2]) Journ. Iron & Steel Inst., 1903, part II, 933.

where it seems that quenching ought to have its maximum effect. The paradox might perhaps be explained by remarking that a too severe chilling breaks the steel, produces expansion and hinders the pressure, one of the most important factors of equilibrium, from giving its full value. But whilst waiting for other information, it is more convenient for the present to suppose that we have to do with a really new constituent.

Prof. Stansfield has also verified the existence of the line Aa, whose position might doubtless be slightly modified, and he concludes from his impact-tests, that this line can serve to define the burnt state. A steel heated a little below Aa is coarse grained and fragile: it is overheated. If it has been heated a little above, the fusion has commenced and the metal is burnt.

But there are degrees of the burnt state. We can have four divisions:

1. If the temperature has only just passed Aa, the liquid is not very abundant and forms small isolated lakes.

2. If the temperature has been high, the liquid tends to form a more and more continuous network round the grains of austenite.

3. If we approach AB, the liquid is partly driven out by the gases which are set free, leaving cavities.

4. Oxidising gases have penetrated into the cavities and have left ferrous oxide there

The burnt steels of the first and second class can be restored by a simple thermal treatment; with those of the third class it is necessary to add mechanical treatment, which, applied with precaution, will enable the cavities to be closed. The steels of the fourth class cannot be completely restored because the oxide remains enclosed.

If during cooling, starting from a point in the region AaB, the carbon, which has accumulated in the liquid, has not distributed itself uniformly in the solid, it produces abnormal islets of cementite in the joints, even with steels which ought not to contain independent cementite.

The thermal treatment thus aims at a double object. It should induce a uniform distribution of carbon and substitute a fine grain for the coarse and dangerous grain produced by overheating. In order to obtain the first result, it is necessary to heat

a little below *Aa*, but this is still overheating. Hence the necessity of a second reheating, this time a little above the broken line *GOSE*, in order to obtain a new grain, the finest possible.

These conclusions resting on micrographic observations and on mechanical tests, are correct. They give an interesting and useful contribution to the theory and practice of annealing.

The most uncertain lines in Roozeboom's diagram are those which outline the regions of graphite.

Tiemann[1]) taking special precaution to avoid the presence of all traces of silicon, has prepared, a white cast iron containing:

Combined carbon	4·271
Graphite	0·255
Silicon	0·000

He reheated it at temperatures between the lines *EFH* and *aBC* (fig. 6.) and quenched the samples, either directly after reaching the maximum temperature, or after having maintained this temperature for an hour. The amount of graphite present was then determined, and it was found that it remained practically constant throughout.

Analogous experiments have been made by Charpy and Grenet[2]) working with a series of castings of nearly constant carbon (3·20 to 3·60 per cent) and of increasing percentage of silicon (from. 0·07 to 2·10). Their conclusions are as follows:

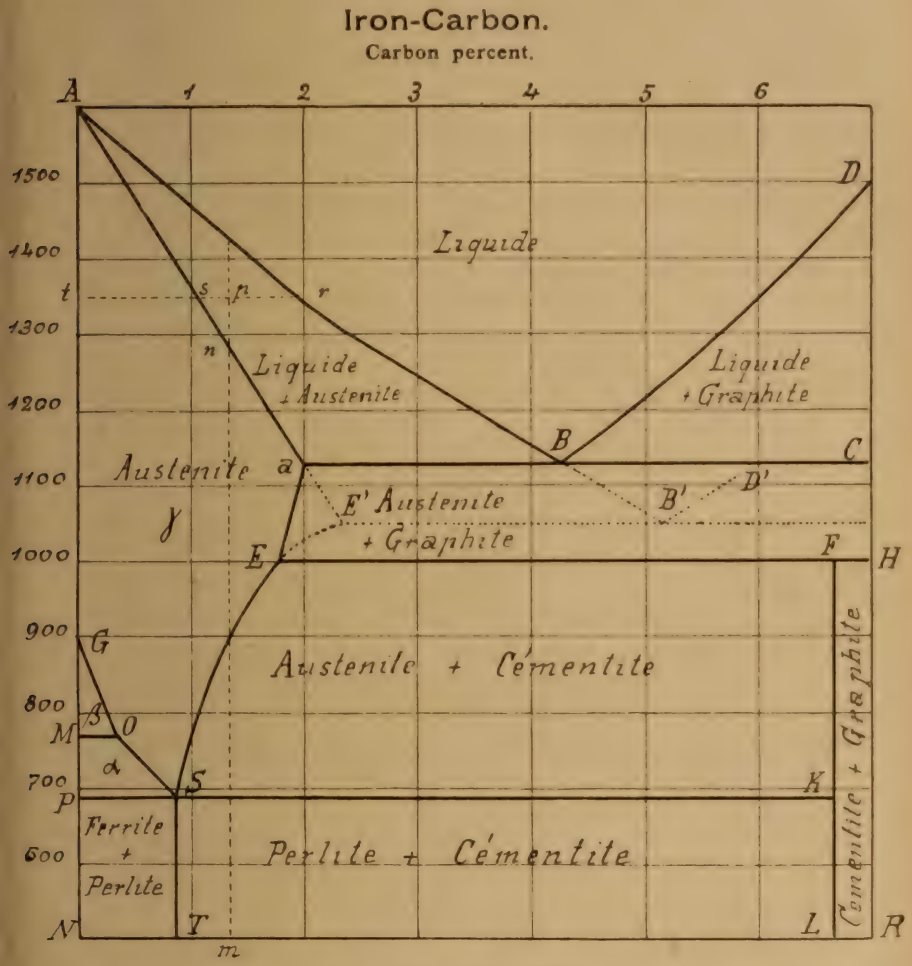
1. The higher the percentage of silicon is, the lower will be the temperature required to bring about the separation of the graphite. Thus in cast iron containing 0·07 per cent of *Si*, a extended heating below 1100° C. did not cause any separation of graphite. With percentage of 1·25 or 2·10, a heating to 650° C. was sufficient to obtain the separation.

2. The separation of graphite once commenced will continue at temperatures lower than those at which the reaction was brought about. Thus a casting with 0·07 per cent of *Si* heated to 1170° C. and quenched, contained 0·50 per cent graphite; another piece heated to the same temperature and cooled slowly to 700° C. and then quenched, contained 1·87 per cent graphite.

[1]) Metallographist, IV, 313, October 1901.

[2]) Bull. Soc. d'Encouragement, CII, 399, March 1902.

3. At constant temperature, the rate of separation of graphite is the slower, the lower the temperature and the lower the percentage of silicon.



Composition by weight.

Fig. 6.

4. The percentage of combined carbon which corresponds to equilibrium at a fixed temperature, diminishes with increasing percentage of silicon.

5. The percentage of combined carbon which corresponds to equilibrium, diminishes with falling temperatures.

We must also mention, as contributions to the study of this question, an article by Charpy[1]) and another by Guillet[2]).

Charpy and Grenet conclude from their results that a certain number of points in Roozeboom's diagram ought to be modified, but it seems necessary to wait for further experiments before proposing to plot a new diagram.

In fact the problem is a difficult one. One has to do with equilibria which are established extremely slowly, and little is known of how the silicon interferes. Its action has been compared with that of a catalytic and is probably very complicated. It is well known that in irons which are almost free of carbon, the silicon seems to suppress point A 3 almost entirely (that is to say, the region of γ iron) when it reaches a percentage of about 2. The carbon seems to oppose this action, but all the same the silicon interferes directly in the equilibria between the allotropic varieties of iron, and these equilibria necessarily determine the state of the carbon. These are the complications which have not yet been accounted for.

Col. Cubillo[3]) has followed up the reaction in the Martin-furnace by means of micrography.

N. Ternary Steels. After the ferro-carbon series, naturally come the ternary systems in which iron and carbon are connected with a third substance, such as nickel, manganese, chromium, tungsten, etc., for the purpose of obtaining special steels.

Works on this subject extended to micrography have been published by Guillet. But as this author will be present at the congress of St. Petersburg, we will naturally leave it to him to the results of his researches.

It remains only to mention, a memoir by J. Matthew[4]) (whose microphotographs are unfortunately too feeble when magnified to be clearly understood), and the thesis by Dr. Böhler[5]), of which we will now give a resumé.

[1]) Comptes rendus Acad. Sc., CXXXVI, 1000, 27 April 1903 and Réunions des membres français de l'Ass. int. méth. d'essai, 30 May 1903.

[2]) Comptes rendus Acad. Sc., CXXXVI, 1319, 2 June 1903.

[3]) The open-hearth process, Journ. Iron and Steel Inst., 1903, part I, 276.

[4]) A comparative study of some low carbon steel alloys; Journ. Iron & Steel Inst., 1902, part I, 182.

[5]) Über Wolfram- und Rapidstahl, Berlin 1903.

It is well known that in tungsten-steels, as in chrome-steels, if the percentage of tungsten or chromium is sufficiently high, the temperature of heating greatly influences the position of recalescence. If the heating is only carried on a little above point *Ac*1 and the steel is allowed to cool, the recalescence will be produced at normal temperature. But the higher the temperature of heating the more does the recalescence tend to descend, either all in one or in two fractions. In steels of suitable composition, it has descended below 400° C. to about 350° C.; the metal remaining hard, i. e. it is self-hardening. This is the theory of the quick steels.

Dr. Böhler has prepared a series of specimens with different percentages of tungsten; he has determined the points of solidification and has proved that the molecular lowering due to the tungsten is the same as that produced by carbon. He has further verified the assertions of his predecessors and has added new information, besides examining the microstructure after quenching between the critical temperatures.

Whatever the previous cooling may have been, the position of the critical point remains constant during heating.

The points of transformation do not ascend again, if the cooling is slower than the normal one.

A prolonged stay at a certain temperature above *Ac*1 is equivalent to a slower heating at a high temperature.

The rate of heating affects the result.

Dr. Böhler concludes that the retarded transformations, single or double, are always of the same nature, that is to say, the transformations of austenite into pearlite or into sorbite.

This conclusion seems well founded, and in our opinion, this is what takes place.

The presence of tungsten or similar substances lowers the percentage of carbon corresponding to the eutectoid. The hard tungsten-steels are thus always hyper-eutectoid, and the cementite in excess is only dissolved progressively during the heating. The metal is divided during heating into two portions: one is of the same percentage of carbon as the eutectoid, the other is of a higher percentage. As a certain time is required for the carbon to be uniformly distributed, the equilibrium is only established if the rate of heating is very slow. Otherwise the division of the metal into two portions of unequal carburation, continues at the

origin of the cooling; the eutectoid portion gives its recalescence at normal temperature. In the region higher in carbon retardation takes place, which increases with the percentage of dissolved carbon.

If the temperature and time of heating have been sufficient to cause a homogèneous distribution of carbon, then there is only one recalescent point at low temperature. This theory, which is supported by unpublished experiments, seems to explain all the observed facts.

III.

Modifications of one isolated phase, or of a complex of phases within their regions. Thermal and mechanical treatments.

In the preceding chapter, we have studied the equilibria which can be established among the phases, but not the dimensions, the forms or the distribution of these phases.

The knowledge of these latter data is nevertheless of great importance, because they have a direct influence on what is of final importance to the engineer, viz: the mechanical qualities of metals and alloys.

A. Structure and modifications of phases isolated in the interior of their region. The pure metals and the alloys formed of a single solid solution chemically homogenous, are however not homogeneous from a physical point of view. They are formed, as has been proved since 1878 by the celebrated work of Prof. Tschernoff, of crystalline grains, each of which is characterised by its size, its shape and its crystalline orientation.

Andrews[1]) has recently added to the numerous known examples, that of platinum. He has caused corrosion figures of the cubic system, analogous to those of iron, to appear on the surface of a polished section by attacking it with aqua regia.

The dimensions of the grains after cooling, depend on the rate of passing from the liquid phase to the solid phase, and on the rate of the cooling of the latter. It seems, and that is also Prof. Howe's opinion, that to each temperature corresponds nor-

[1]) Microcrystalline structure of platinum; Proc. Roy. Soc., LXIX; also Metallographist, V, 233, July 1902.

mally a certain size of grain and which would be obtained the mere slowly, the lower the temperature is; but it can never be obtained if the temperature is too far from the fusing-point. Howewer in the pure metals and in the homogeneous solution, it is not at all certain that the heat treatment alone can change the aquired structure during cooling. The grain of the tin on tinned iron, which is made fine by quenching, does not become coarser by annealing (Ewing and Rosenhain).

Opposite facts might be cited. Thus Stead[1]) has annealed soft finely granular iron for several years, and has caused the grain to grow to such an extent, that one of the new grains may represent the union of 4,000.000 of original ones. Beilby[2]) has shown that at temperatures much lower than the freezing-point, the molecules have sufficient mobility to displace and group themselves under the action of surface tension.

But these examples are not very convincing, because one can always, if not prove, at least suppose, the presence of traces, indeterminable or not determined, of some foreign material.

According to Ewing and Rosenhain[3]) a previous deformation is necessary, in order that a re-crystallisation can be produced during annealing. It is at least certain that it is a sufficient condition. These authors have tried cadmium, lead and zinc. Annealing is produced spontaneously in lead at ordinary temperature. The new grain grows quicker and more when the temperature approaches nearer to the fusing-point. Otherwise there is no proof that the size of the grain is a function of the temperature. The new grain is often characterised, especially in lead, by the presence of macles which did not exist in the cast metal.

Lead, cadmium and zinc do not appear to have critical points in the solid state. Ewing and Rosenhain think, while giving their explanation as a supposition, that the growth of the grain by annealing is the result of the dissolution of the metal in an eutectic placed between the grains, and of its consecutive precipitation, in consequence of the unequal solubility of the differently oriented

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[1]) Memoir cited further on.

[2]) Proc. Roy. Soc., LXXII, 226; also Revue de métallurgie. I. abstracts, 33, January A R S U 1904.

[3]) The crystalline structure of metals; Phil. Trans. Roy. Soc. CXCV, 279; also Metallographist, V, 81, April 1902.

faces of two neighbouring grains. And it seems probable that this phenomenon is a form of electrolysis. When one does not see any eutectic, one can always imagine one, invisible and present; it is not even necessary that this eutectic should be liquid at the annealing temperature, if there is solubility at the solid state, and if the molecular migration is possible at the considered temperature.

In supporting their theory, Ewing and Rosenhain[1]) have at any rate made some very pretty experiments. If two freshly cut lead surfaces be welded by pressure, the annealing will cause a re-crystallisation owing to the deformation necessitated by the weld, but the line of the weld remains clear, and a grain never extends from the one half into the other half. Let us now introduce into the weld a film of a foreign substance capable of forming a readily fusible eutectic; the weld remains still perfectly clear, if the eutectic film is continuous; but if this film shows any discontinuities, one obtains, by annealing, grains which traverse the line of the weld at the place of these discontinuities; electric currents resulting from the difference of potential between the contiguous and differently oriented grains have now become possible. Cold-hammering, on this supposition, would only serve to break up the eutectic.

J. C. W. Humfrey has made similar researches, but with specimens formed of an unique lead crystal. He found that the planes of deformation (or slip planes) are parallel to the four pair of faces of a regular octahedron. This is the same process of crystalline deformation which Mügge has shown for copper.

The division by deformation of the large original crystal into small crystals of varied orientation is not instantaneous. An examination immediately after deformation only shows the division in regions which are sufficiently deformed; for example, on the side, of a sheet bent and straightened out again which has been first submitted to compression. But the division into small grains extends afterwards to less deformed regions, spontaneously at ordinary temperature and quicker at higher temperatures.

W. Campbell[2]) has made similar observations with tin.

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[1]) Effects of strain on the crystalline structure of lead; Phil. Trans. Roy. Soc., (A), CXCIII, 353 and CXCV, 279; also Metallographist, VI, 250, July 1903.

[2]) Crystallisation produced in solid metals by pressure, Metallographist, V, 57, January 1902.

These facts, which come near to those found by Charpy with brass, and by Heyn with copper, seem to prove, we think, that the new grain is always the effect of annealing, spontaneous or otherwise, not of deformation. The deformation would only destroy the existing equilibrium and would bring the metal into a metastable condition; the annealing would restore a new equilibrium in connection with the new conditions, while taking into account, of course, certain local crystalline orientations of deformed material.

B. Modification of a complex of phases in its region. Just as, in a solid formed of a single phase, the size and shape of the crystalline grains depend on the conditions in which this phase has been constituted, starting from the previous phase, so, and for stronger reasons, in a complex, the size and the form of both phases and their mutual distribution depend on the conditions in which these two phases have been separated and cooled.

The distribution can be extremely variable, from emulsion closely allied to dissolution, up to complete separation into nuclei and envelopes.

In chapter II we have given several examples of heterogeneous distributions belonging to the copper-silver, copper-iron and copper-antimony series. According to the phase rule, however, the equilibrium was probably established, but the constituents of the same nature require a sufficient time and a sufficient temperature to gather into elements of homogeneous structure micrographically visible.

We have thus obtained a notion of a structural equilibrium different to the physico-chemical equilibrium, an equilibrium which s actually found when the phases have been individualised in the most complete manner possible, and have acquired their maximum dimensions.

If the conditions of cooling have not permitted the complex to attain this equilibrium, it may often be approached either by thermal treatment only or by thermal and mechanical treatments combined.

We owe to Stead[1]) an important work on this question of which we will attempt to make an abstract, without entering

[1]) The segregatory and migratory habit of solids in alloys and steel below the critical points, Journ. Soc. Chem. Industry, XX, 31, 18 March 1903.

upon the more difficult details relative to certain ternary alloys. Stead, considering the thermal treatment, calls coalescence the process by which the molecules of the same phase tend to approach each other, and to gather together into structural elements of the largest possible size.

The coalescence within a region is naturally so much the easier and so much the quicker as the temperature is maintained nearer the higher limit of the region, and as the phases about to separate are in more widely differnig relative proportions.

For example with the eutectoid alloys ferro-carbon, the physico-chemical equilibrium is settled directly after recalescence, if the cooling be slow enough for the transformations to be complete. But the cementite is at first infinitely divided in the α iron and if the cooling be accelerated immediately after the transformation, the coalescence does not take place. A constituent is then obtained which in appearance is homogeneous and resolvable under the microscope at very high magnifications, and to which one of us has given the name of sorbite, in honour of Dr. Sorby, the first pionneer of microscopic metallography. By means of a slower cooling, the ferrite and cementite individualises at first in alternating laminae more or less fine: it is the pearlite, the most usual form of eutectics and eutectoids. When the rate of cooling is still slower, the cementite individualises still better, and at the limit tends to form cellular envelopes round the grains of the ferrite.

Whatever may be the structural distribution at which one has arrived spontaneously, one would generally be able, if this distribution is far enough from the equilibrium, to modify it by an annealing in its region.

The distribution of the phases is of great practical importance. The sorbite offers a high resistance to rupture with minimum brittleness. The extra cellular distribution of the cementite, added to the crystallisation of as many better developed nuclei of ferrite, produces the maximum brittleness.

There is still another manner of distribution of the two phases ferrite and cementite; the cementite is disseminated into straggling spherulites more or less large and scattered. This structure might be the result of a deformation continued during and a little below recalescence.

Lange[1]) attributes to it, which is very probable, the maximum ductility compatible with the percentage of carbon; that does not mean minimum brittleness.

Lau[2]) recommends this same spherulitic structure for tool-steels which are to be used chilled. In this case, it probably prevents, when heated somewhat quickly, the homogeneous distribution of carbon, and thus leaves after quenching a network relatively soft which will prevent bending and will give body to the tool.

C. Indirect modification of a complex of phases. — The distribution of the phases in the final complex, which is made use of in engineering structures, does not only depend on the thermal and mechanical treatments which have been gone through in the region of this complex, but also on the corresponding treatments gone through in the previous regions. The passages through these various regions can leave, and generally do leave, traces in the structures and final properties.

With all metals originally cast, the process of solidification is of importance, and the more so, the less the metals are mechanically treated afterwards. Longmuir[3]) has studied the micrographic and mechanical consequences of the casting temperature on cast iron, gun metal and brass. The author concludes that for each of these alloys there exists a best casting temperature, which ought neither to be too high nor too low. These results are of practical interest for the foundry.

With steels, it is necessary to be careful not only about the conditions of solidification but also about the passage through the region of the austenite. Theoretically according to the phase rule, the final state ought to be independent of this passage, provided that the transformations have had time to be finished. But it is not the same thing, if we make allowance for the structural equilibria. In fact it has been settled beyond doubt, that the heat treatment in the region of the austenite has a considerable reverberating effect on the final mechanical properties, and to such a degree, that in most works on annealing, it is the only thing which one troubles about. It is probable that the structure of the com-

[1]) Metallographist, VI, 9, January 1903.
[2]) Metallographist, VI, 322, October 1903.
[3]) Journ. Iron and Steel Inst., 1903, part I, 457.

plex ferrite + cementite and especially the crystallisation of the ferrite, grow into one substance in consequence of the previous condition of which they are the responsible successors.

There are two problems in practice which should be specially considered, viz:

a) To restore the defective steels by a heat treatment;

b) To prevent the steels from becoming defective by a skilful mechanical and thermal treatment.

Metallography is very useful and has been employed as an auxiliary for studying this question; its work is to show and to record the characteristic structure due to a given treatment applied to a given steel.

The types once settled, the structure allows one to recognise the treatment which it has undergone, and which was supposed to be unknown.

a) From the point of view which we take, steels may be divided into cast steels, and rolled or forged steels. The cast steels, after simple cooling, are always unfit for use: it is customary to use a thermal treatment. With rolled steels it may be necessary, useful, or harmful, according to circumstances. It will be useful if the rolling has been terminated at too high a temperature; but then the case is more or less like that of castings.

In giving an abstract of Prof. Stansfield's researches, we said that the thermal treatment applied to cast or overheated steels should destroy the bad structure and substitute a better one for it. These are two operations in one. In order to destroy the bad structure which has been acquired in the region of the austenite, it is necessary to return into this region, sufficiently far to efface the bad, and not far enough to have a relapse. This is a problem which may be difficult, and up to now, it was perhaps wrong not to divide it.

During the period which we have just reviewed, W. M. Carr[1]) has made annealing of castings the subject of some researches.

Annealing of rolled steels has also been the subject of several interesting studies.

[1]) Annealing of steel castings, Metallographist, V, 58, January 1902.

Prof. Heyn[1]) has made some methodical researches and has not only taken into account temperature but also time. He has plotted his results as a surface with the following three co-ordinates: heating-time, annealing-temperature and capacity for pliability, the latter being results obtained from impact-bending-tests on notched bars. This introduction of fragility as a criterion of the effects of annealing is a happy innovation. It is stated that fragility can appear in the extra soft steels at relatively moderate temperatures, if heating is sufficiently prolonged. Such a steel, very pure and annealed to 1100° C., becomes more and more fragile when the annealing has gone beyond 6 hours. An annealing of 14 days between 700° and 800° C. did not increase the fragility.

The size of the grain is not necessarily a sign of fragility, or vice versa.

Stead and Richards[2]) have paid special attention to the restoration of overheated rails; a simple reheating towards 900° C. followed by a rather rapid cooling, suffices to obtain a metal of good quality. These authors have used Wöhlers method (small alternate dresses) to test the effects of the treatment.

We would also mention, on the same general subject, a memoir by Campion[3]) and a note by Lloyd[4]).

b) Instead of restoring the badly treated steel, it is simpler to use a well directed treatment. The road leading to this object has been opened up by Prof. Tschernoff who in his memorable memoir of 1868 has laid down the rules, on their true basis, for working steel and which can still assist the always growing development of his work.

As long as steel is submitted red hot to mechanical processes, the crystallisation is always broken off and cannot be organised.

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[1]) On the overheating of mild steel. Journ. Iron and Steel Inst., 1902 part II, 73.

[2]) On the restoration of dangerously cristalline steel by heat treatment, Journ. Iron and Steel Inst., 1903, part II, 119.

[3]) The heat treatment of steel under conditions of steelworks practice, Journ. Iron and Steel Inst., 1903, part I, 378.

[4]) Note on the heat treatment of steel rails high in manganese, Journ. Iron and Steel Inst., 1903, part II, 353.

One must terminate the work at a somewhat low temperature, which Tschernoff calls point *b* and below which the grain does not grow appreciably, even during slow cooling.

But the fever for large productions at low prices has caused the steel-works to neglect these wise rules, and to consider the rolling as a purely mechanical operation, finishing the bars at higher and higher temperatures. Hence a deterioration of the produce.

A. Sauveur, guided by Prof. Howe's experience, has been amongst the first to conclemn this regrettable practice. He presented a very excellent paper to the Chicago Congress in 1893, in which metallography furnishes a numerical measure of the size of the grain. Since then he has found numerous followers, chiefly in America. The Kennedy-Morrison and other systems seem to owe their origin to this cause. Among recent papers and articles on this subject, we may mention those of S. S. Martin[1]) and Job[2]). Martin criticises the Kennedy-Morrison process, which consists, as is well known, in leaving the rail to itself during one or two minutes before placing it in the last grooves. While remaining at this too high temperature, the steel will still easily crystallise and the final rolling will only destroy this defective structure superficially. It would be better to finish at about 850° C. without any rest.

By means of metallography and impact-tests, Job examined a number of rails which had done bad service, and a number of good rails. The fragility and want of resistance to wear and tear were due to coarse grain.

Le Chatelier[3]) believes that the rolling might be finished during recalescence. On account of the heat given off, the temperature will be maintained constant during a sufficiently long time, and one has thus a natural mark, easy to find, even in manufacturing.

[1]) Rail rolling at lower temperatures during 1901, Metallographist, V, 191, July 1902.

[2]) Relations between structure and durability, Metallographist, V, 177, July 1902.

[3]) Bull. Soc. d'Encouragement, CIII, 394, September 1902.

It is however necessary to find out whether our rolling mills are fit for this procedure. Generally the temperature of the final pass is much higher than the recalescence, and it may be expedient, if one wishes to realise the sorbitic stage, to accelerate the cooling artificially before and during the passage through the critical points.

The memoir of Stead and Richards[1]) on sorbitic rails embodies this idea. Sauveur and Boyton[2]) have come to the same conclusions.

Dudley[3]), after visiting the principle manufacturing centres in the United States, has drawn up a report giving a complete idea of the state of rail-rolling in his country.

We also wish to mention an article by C. Mercader[4]).

IV.

Divers questions.

A. Chemical deterioration. In constructions, the mechanical deteriorations are usually more rapid than the chemical ones, hence the legitimate preponderance of mechanical tests. But this is not always the case. In naval constructions, for instance, certain portions, which are in contact with sea water may corrode rapidly.

J. T. Milton and W. J. Larke[5]) have gone into the questions of chemical deterioration and of the choice of alloys to oppose them. They have borrowed the assistance of metallography with success.

As is well known, the gray phosphoric cast iron is strongly attacked by sea water, which converts it into a spongy mass without any consistency. The alteration originates from the graphite and leaves the phosphoric eutectic alone.

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[1]) Journ. Iron and Steel Inst., 1903, part II, 141.

[2]) Metallographist, VI, 148, April 1903.

[3]) The rolling and structure of steel rails. Metallographist. VI, 111, April 1903.

[4]) Hollow pressed axles, Journ. Iron and Steel Inst., 1903, part I, 95.

[5]) The decay of metals, Proc. Inst. Civil. Engs., paper No. 3424.

In the tubes and plates of the condenser, which are always in contact with sea water, the Müntz-metal becomes more and more impoverished in zinc. Adopting the ideas of Prof. Arnold, the authors think that the two constituents of the alloy form galvanic elements of which the one loses zinc until the poles are reversed. The second constituent then commences to give off zinc in its turn; thus there are two stages in the destruction. The metal remains yellow during the first stage and becomes red during the second one; at the end it is only a mixture of copper and zinc oxide. This is confirmed by microscopic examination.

The addition of a little tin to the alloys acts as a very marked preservative, as already shown by the trials instituted by Lloyd's Register.

Gun-metal resists sea water better than brass. The change appears to begin with the envelopes of the crystallites, which, while remaining metallic, change to a darker hue.

Besides uniform deterioration, rapid local perforations have also been noticed, for instance, in condenser tubes made of homogeneous brass. These perforations may be due, either to local accumulations of impurities, or to mechanical erosions starting from irregularities of the surface. It is important that the surfaces should be well polished.

The authors also recommend, for metals and alloys exposed to sea water, an insulation as complete as possible of all the electrical cables in the neighbourhood.

B. The effect of impurities on commercial copper, by E. A. Lewis, Engineering, LXXVI, 753; also Revue de Métallurgie, I, abstracts, 69, February 1904.

The impurities of copper may be divided into three classes according to micrographic examination.

1. Metals which from round the crystalline grains of copper, such as lead and bismuth.

2. Metals which alloy with a certain portion of the copper and from round the copper grains, such as arsenic, antimony, tin and zinc. The non-metals oxygen, phosphorus and sulphur behave in the same manner. This group may be subdivided according to whether the alloy separates out into two constituents or not. Antimony, zinc and oxygen are thus separated; arsenic, phosphorus and tin form solid solutions.

3. Metals which form with copper a homogeneous solid solution, such as iron, manganese, aluminium and nickel.

The fact that the presence of 0·2 per cent of zinc will give an eutectic, is in contradiction with previous researches.

Arsenic neutralises to a certain extent the bad effects of lead and bismuth by permitting the dissolution of these metals.

C. Determination of cuprous oxide in copper by micrographic analysis. H. O. Hofman, C. F. Green and R. B. Yerxa, Trans. Amer. Inst. Min. Eng., New York Meeting. October 1903.

It is sufficient to measure the surface of the eutectic by means of a planimeter on a polished section. The results are in accord with those of chemical analysis, and they are obtained much more easily.

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37 e (Suppl.)

INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 37.

Group F: Metals

REPORT ON THE PROGRESS OF METALLOGRAPHY SINCE THE BUDAPEST CONGRESS 1901.

(SUPPLEMENT.)

BY **F. OSMOND**, PARIS.
(TRANSLATED BY CH. CAPITO, LONDON.)

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Report on the Progress of Metallography since the Budapest Congress 1901

(Supplement).

By **F. Osmond**, Paris.

(Translated by Ch. Capito, London.)

The St. Petersburg Congress having been postponed, necessitates the publication of this supplement. Since the commencement of 1904, the development of metallography has been rapid. New centres of studies have been created, amongst which the laboratory of Prof. G. Tammann at Goettingen is now one of the most active. It becomes more and more difficult to keep exactly in the current, and the writer apologises to those authors whose publications he may have ignored.

The plan of this supplement is the same as that of the initial report.

Baron de Jüptner[1], with incessant zeal, has published his «Grundzüge der Siderologie» of which the third part appeared in 1904, while the first part has been translated into French as «Etude théorique des alliages métalliques»[2].

Mr. L. Guillet has written a treatise which is more summary and less specialised, but inspired by the same principle, the connection between metallurgy and physical chemistry

To these didactic treatises, we must add a paper by Prof. Tammann[3] in which a marked progress of the interpretation of

[1] Arthur Felix, Leipzig 1904.

[2] Dunod, Paris 1904.

[3] Über die Anwendung der thermischen Analyse in abnormen Fällen, Zeitschr. anorg. Chem., XLV, 24. (1905).

the curves of equilibrium is shown. In the abnormal cases, which are in reality the most frequent, where the existence of a definite compound is not revealed by a maximum of the curve, its existence may be proved by a maximum arrest, during cooling, in the eutectic curve. Many diagrams previously constructed ought to be revised so as to make use of these new data.

Metallographers who are anxious to obtain definite results, would also profit by the very judicious observations which Mr. E. S. Shepherd[1] brings to their notice. When tracing a curve of equilibrium, it is necessary to constantly agitate the alloy in presence of germs to avoid surfusions, and to assure oneself, by a metallographic examination or otherwise, that equilibrium has been established. If this is not the case, one must try to obtain it by sufficiently prolonged annealing, sometimes for weeks, etc, at an appropriate temperature. For want of these precautions, inexact interpretations have often been suggested, and certain work, too premature, has ended only in loss of time and energy.

I.

Progress of technical metallography.

A. Preparation of polished surfaces. Mr. G. T. Beilby[2] has continued his experiments in the case where polishing alters the surface of solids, and finishes up with a general theory of deformation founded on the possible existence of several solid phases, of which one is thermally and another mechanically stable, without counting the intermediate phases. This is not the place for discussing this theory. What we must remember, from a practical point of view, is that polishing creates by itself, if it does not already exist, a superficial hardened layer, which it is always useful and sometimes necessary to get rid of when one is studying the progress of deformation. Messrs. Osmond and Cartaud[3] have studied the processes which must be followed to obtain this result in any particular case.

[1] Iron and Steel Mag., VIII, 222, Sept. 1904.

[2] The surface structure in solids, Journ. Soc. Chem. Industry, 15 Nov. 1903; the hard and soft states in metals. A paper read before the Faraday Society on June 9, 1904.

[3] Les enseignements scientifiques du pallissage. Revue gen. Sciences pures et appl. XVI, 51 (15 Jan. 1905).

If one wishes to get rid of the superficial hardened layer without too much trouble, it is best first of all to avoid the formation as much as possible. The Americans, as described by Mr. W. C. Smeaton[1], always urge that this object is obtained by turning the polishing discs at a very high speed. It is also best to press lightly against the disc, to prevent a deep surface-deformation of the specimens.

Mr. H. Le Chatelier,[2] who has undertaken to simplify all the operations of metallography so as to make this subject more generally useful and to put it within reach of all those interested, uses as polishing powder, firstly «2 minutes» emery, then emery-dust washed in a continuous apparatus; finally alumina obtained by calcination of alum, powdered in a mortar and washed according to Schloesing's process. The powder is spread out on a piece of flannel or on a piece of fine cloth impregnated with soap, or it may be projected with water by means of a pulverizer.

B. Re-agents. For steels, Mr. Smeaton recommends a solution of two per cent of nitrate of ammonium at a temperature of 40°. Mr. Le Chatelier recommends for steels the following two re-agents due to Mr. Kourbatoff:

1° Solution of 4 per cent of nitric acid in isoamylic alcohol.

2° One part of a solution of 4 per cent nitric acid in ordinary alcohol, mixed with three parts of a saturated solution of nitrophenol in ordinary alcohol.

The choice of these re-agents depends upon the degree of molecular association of the dissolvent.

The use of alcoholic picric acid, as already mentioned, has become very general.

D) Photography. Mr. Le Chatelier now uses the Nernst lamp instead of the mercury lamp; he has made his camera horizontal, and has adapted the ordinary stage to his microscope.

For all technical details it is necessary to consult the original papers.

---

[1] Notes on the Etching of Steel Sections. Iron and Steel Mag., IX, 222, March 1905.

[2] Sur la technique de la métallographie microscopique. Revue de Métallurgie, II, Mém., 528, 1905.

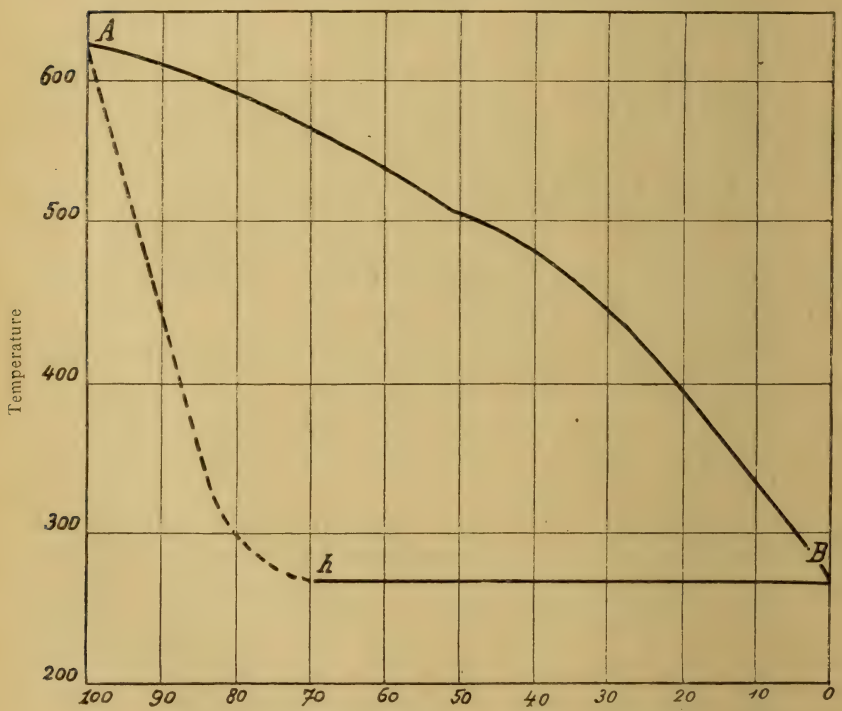
II.

Curves of equilibrium.

A) Antimony and bismuth. K. Hüttner and G. Tammann, Zeitsch. anorg. Chemie, XLIV, 131, 1905.

Roland-Joselin[1] has found that these two metals mix isomorphically in all proportions. This conclusion is perhaps correct but equilibrium is only established very slowly, so that the curves vary with the speed of cooling. If the cooling is not sufficiently slow, mixed crystals of variable eomposition are deposited which are finally of almost pure bismuth in the case of alloys rich in bismuth. (Fig. 1.)

Antimony-Bismuth (Hüttner and Tammann).



Composition by weight: Per cent of Antimony.

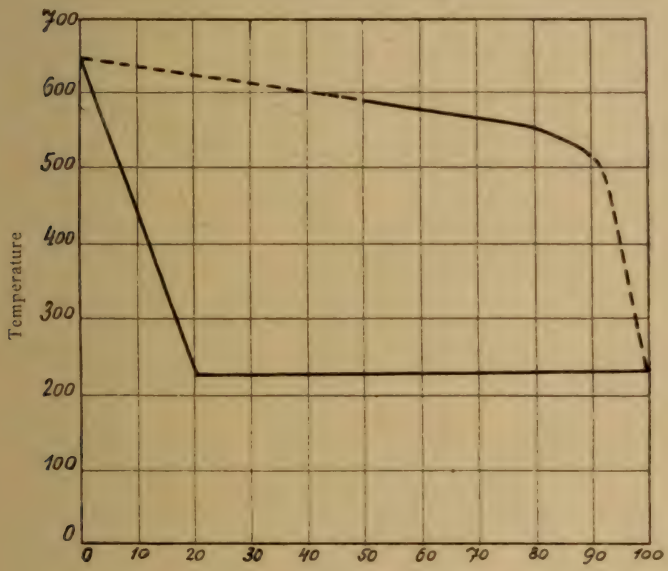
Fig. 1.

[1] Bull. Soc. Encouragement, 5, I, 1293 (Oct. 1896).

B) Aluminium and tin. E. S. Shepherd, Journ. Physical Chemistry, VIII, 233.

Gautier[1] found on the fusibility-curve of alloys, a maximum which corresponds to the definite compound Al_3Sn, with which Messrs. Campbell and Mathews[2] agree. Mr. Guillet thought that he had isolated this compound as well as another one of the formula Al_4Sn[3] by purely chemical processes from ingots reduced by alumino-thermy.

Tin-Aluminium (E. S. Shepherd).



Composition by weight: Per cent of Tin.

Fig. 2.

Finally Messrs. Anderson and Lean pointed out a horizontal portion in the liquidus-curve which would suggest the presence of two superposed liquid layers.[4]

In view of these contradictions, Mr. Shepherd renewed the investigation of this subject. The curve has no horizontal

[1] Bull. Soc. Encouragement, 5, I, 12, 1293 (Oct. 1896).

[2] Journ. Amer. Soc., XXIV, 253, 1903

[3] Comptes-rendus Ac. Sc., CXXXIII, 935, 1901.

[4] Proc. Roy. Soc., LXXII, 277, 1903.

portion. There is produced neither conjugated liquids nor definite compounds: the existence of a solid solution has been established, the saturation of which corresponds to 20 per cent of tin.

C) Gold and nickel. Mr. Levin, Zeitsch. anorg. Chemie, XLV, 238, 1905.

This case is very simple: no definite compounds; the two metals possess a very marked mutual solubility. Eutectic C (Fig. 3)

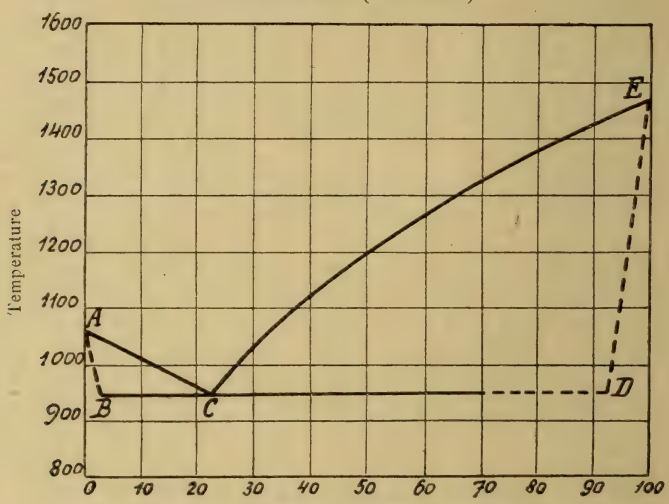


Fig. 3.

D) Gold and thallium. Mr. Levin, Zeitsch. anorg. Chemie, XIV, 31, 1905.

These two metals do not form a definite compound and are mutually insoluble. Eutectic at 131 degrees. It is possible that thallium undergoes two transformations at 225 and 180 degrees respectively. The corresponding liberations of heat are extremely feeble. (Fig. 4.)

E) Magnesium and tin. G. Grube, Zeitsch. anorg. Chemie, XLVI, 76, 1905.

A definite compound $Sn Mg_2$; two eutectics, *B* and *D* (fig. 5); absence of solid solutions.

F) Magnesium and lead. G. Grube, Zeitsch. anorg. Chemie, XLIV, 117, 1905.

The same as in the preceding case. (Fig. 6.) A compound $PbMg_2$.

G) Magnesium and aluminium. G. Grube, Zeitsch. anorg. Chemie, XLV, 225, 1905.

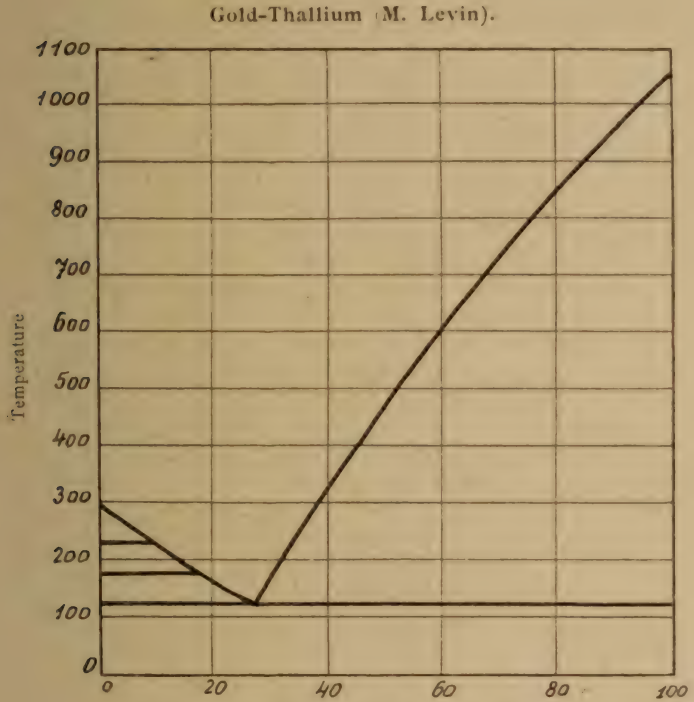


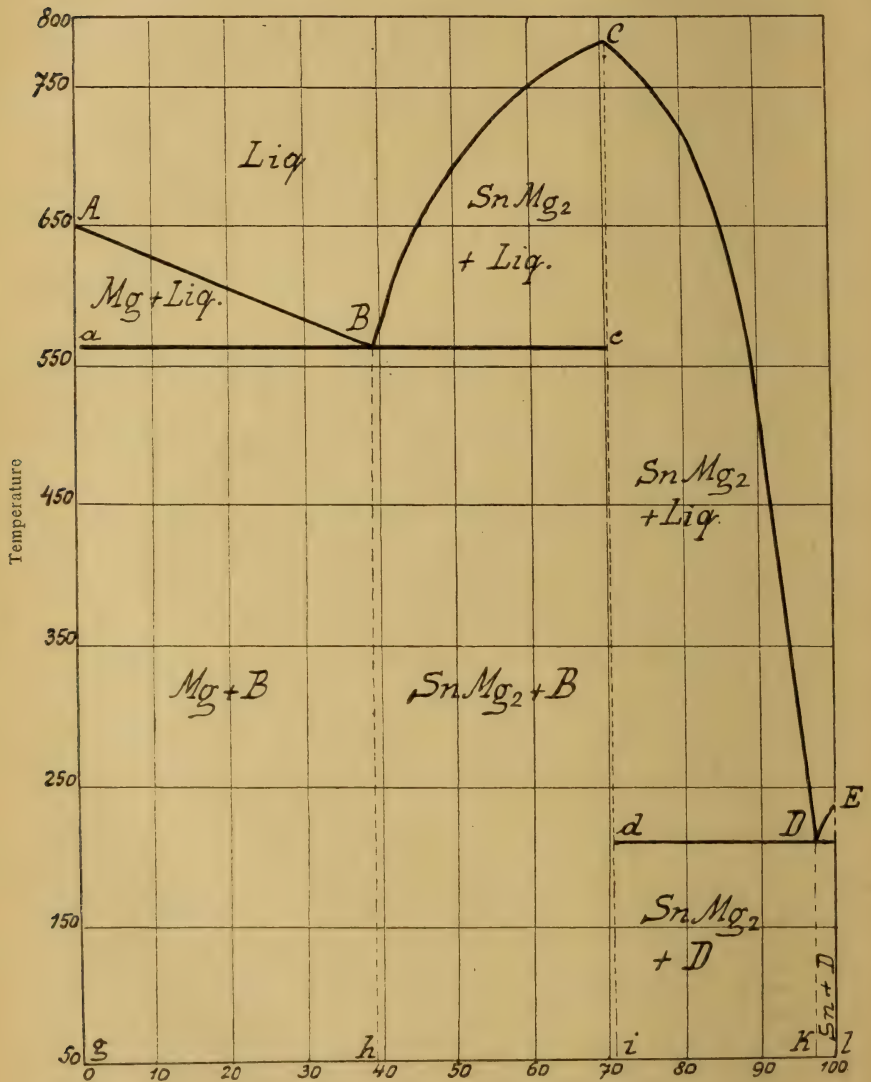
Fig. 4.

The discussion of the results leads to conclusions which are different to those arrived at by Mr. Boudouard, who points out three definite compounds: Al_4Mg, $AlMg$, $AlMg_2$. Mr. Grube recognizes only one single compound, to which he assigns the formula Al_3Mg_4. Two eutectics, *B* and *D*. (Fig. 7.) The eutectic *B* does not occur below the line *B C*: in this region, it appears to form mixed crystals of *Al* and Al_3Mg_4.

H) Magnesium and zinc. O. Boudouard. Rev. Métallurgie, I, Mém. 545, 1904.

The author has only taken the fusibility-curve. (Fig. 8.) The maximum points to the compound Zn_2Mg only; but the chemical and micrographical analyses reveal another, viz: $ZnMg_4$.

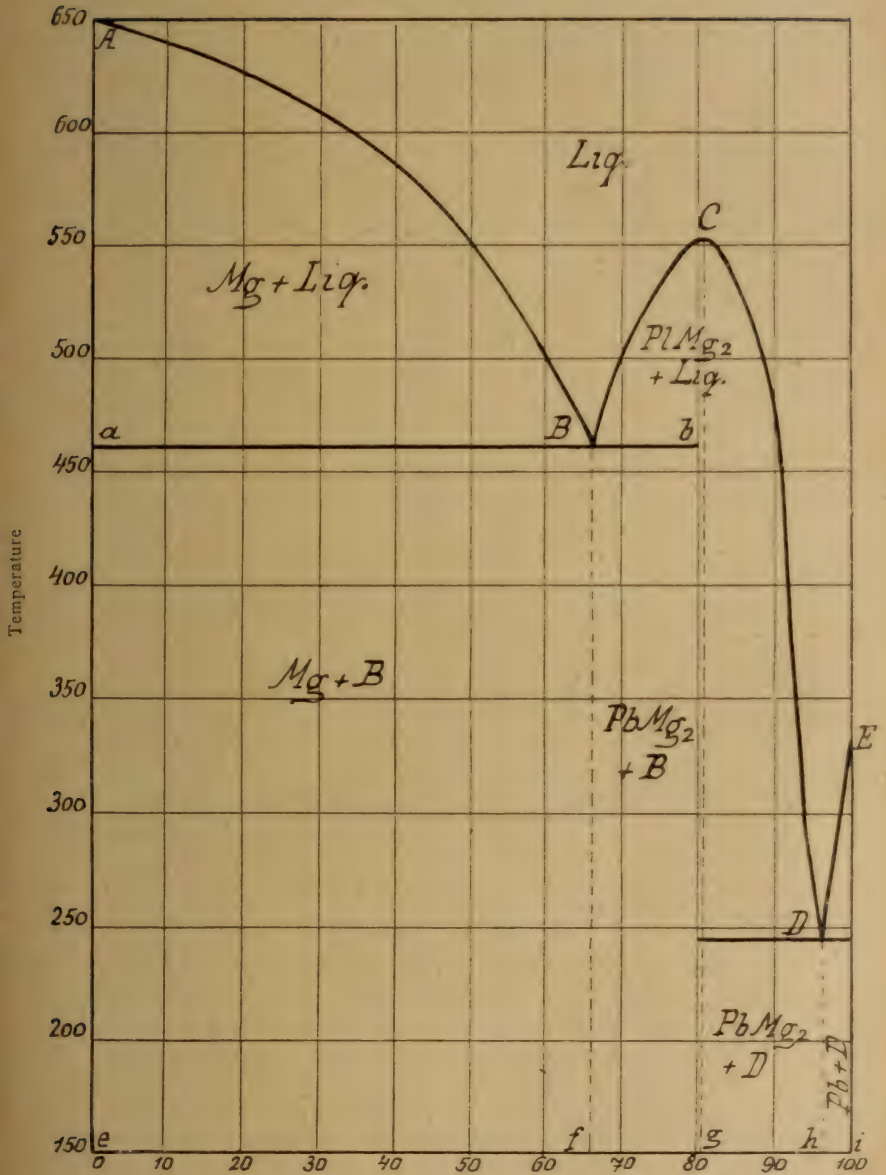
Magnesium-Tin (G. Grube).



Composition by weight: Per cent of Tin.

Fig. 5.

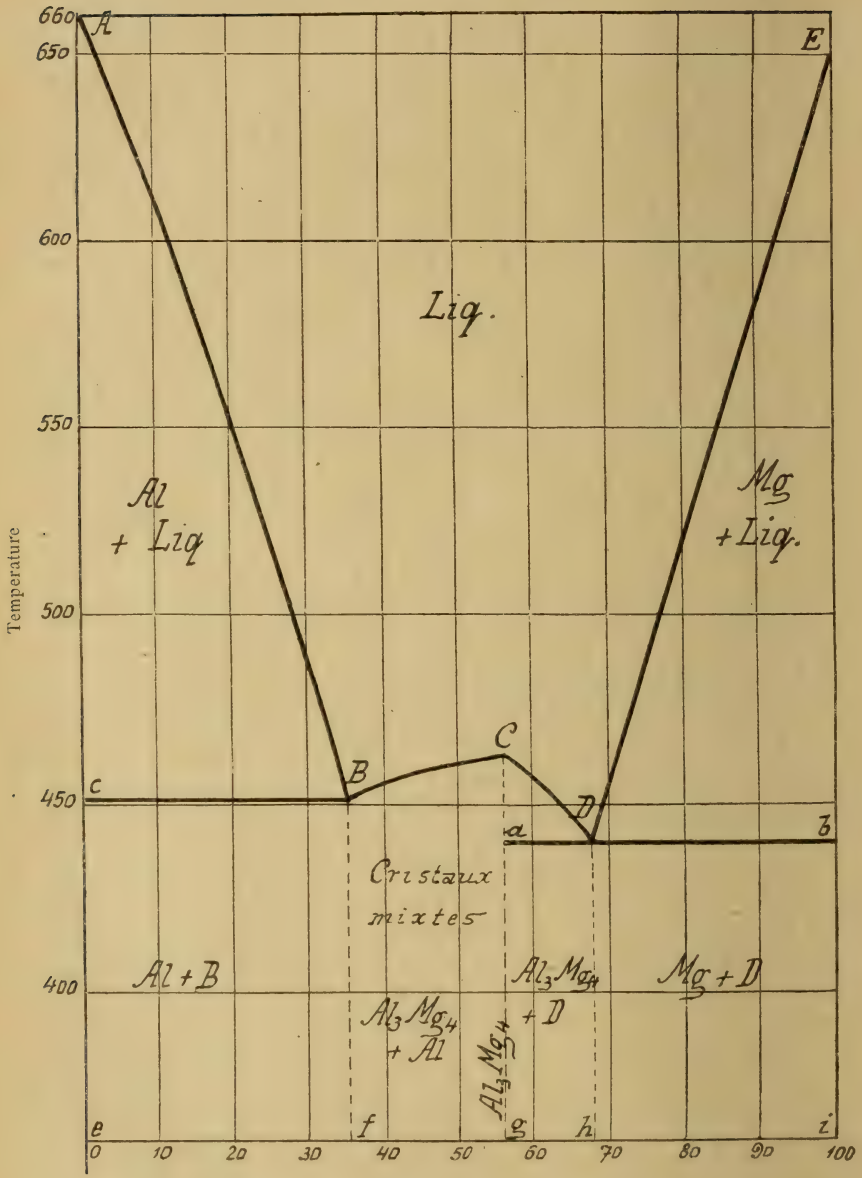
Magnesium-Lead (G. Grube).



Composition by weight: Per cent of Lead.

Fig. 6.

Magnesium-Aluminium (G. Grube).



Composition by weight: Per cent of Magnesium.

Fig. 7.

Cristaux mixtes = mixed crystals.

l) Tellurium and bismuth. K. Mönkemeyer, Zeitsch. anorg. Chemie, XLVI, 415, 1905.

The curve is still of the same type, as the four preceding. A compound: Bi_2Te_3; two eutectics *B* and *D* (fig. 9); no mixed crystals.

k) Zinc and antimony. Mr. Mönkemeyer, Zeitsch. anorg. Chemie, XLIII, 182, 1905.

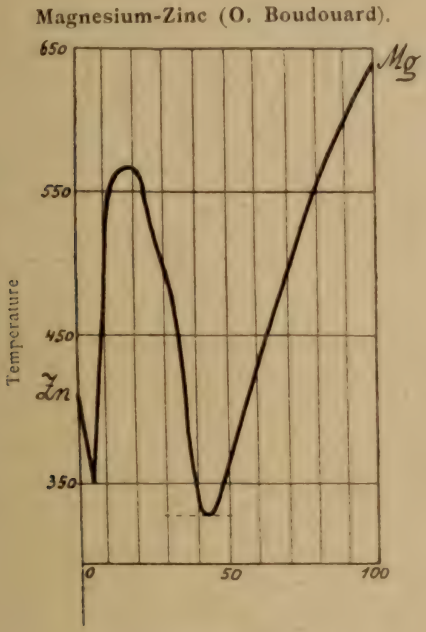


Fig. 8.

The fusibility-curve determined by Mr. Roland-Gosselin[1] seemed to show two definite compounds Zn_3Sb_2 and another containing about 85 per cent of zinc.

Mr. Mönkemeyer does not find the latter, whereas, he confirms the existence of Zn_3Sb_2 and discovers $ZnSb$ (Fig. 10). The diagram shows three eutectics.

The zinc is dimorphous, as Mr. Le Chatelier has already shown by another method[2] and is transformed at 321 degrees.

[1] Bull. Soc. Encour., 5, I, 1293 (Oct. 1896).

[2] Comptes-rendus Ac. Sc., CXI, 454, 1890.

Zn_3Sb_2 is also dimorphous and is transformed at 359 degrees. We ought therefore to have two horizontal lines one for each of these two temperatures and between 45 and 100 per cent of zinc. In reality these two horizontal lines are reduced to one single inclined line *H I K*, probably because Zn_3Sb_2 dissolves a certain quantity of zinc. Likewise, *ZnSb* dissolves a little antimony.

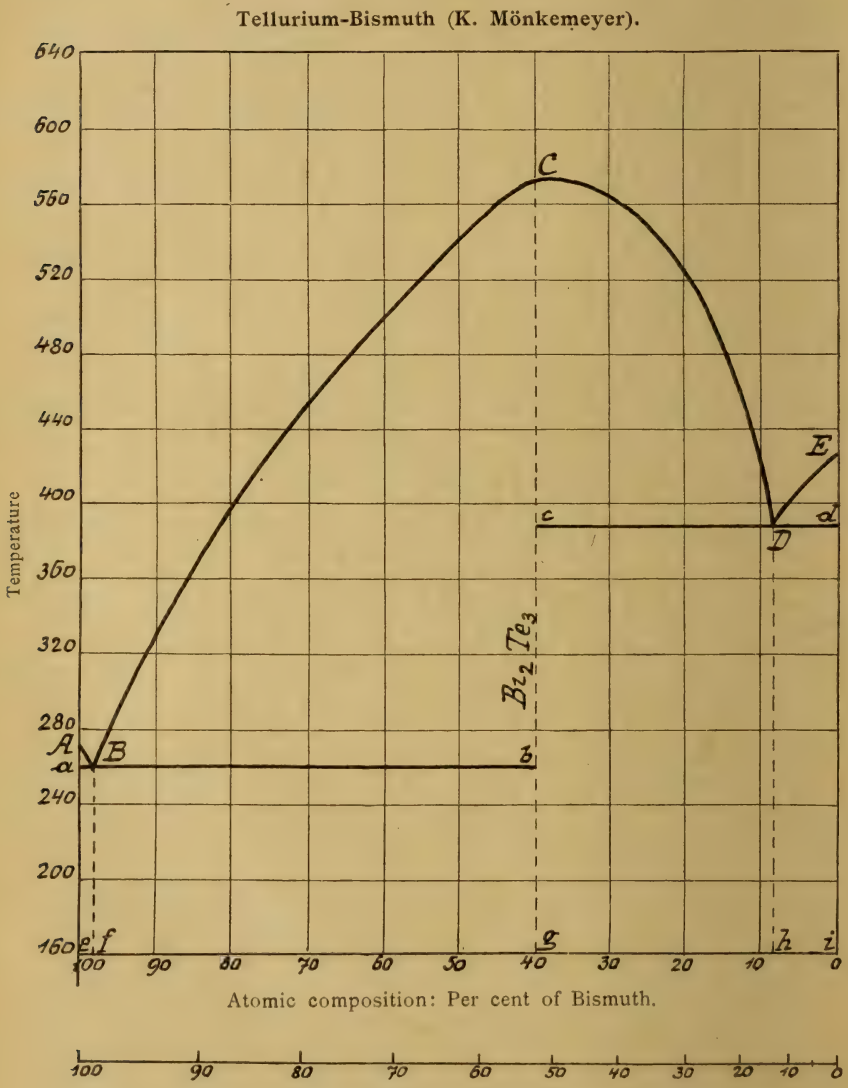


Fig. 9.

L) Tin and antimony. W. Reinders, Proc. Mech. Eng., 209, 1904, also Revue de Métallurgie, I, extraits, 929, 1904.

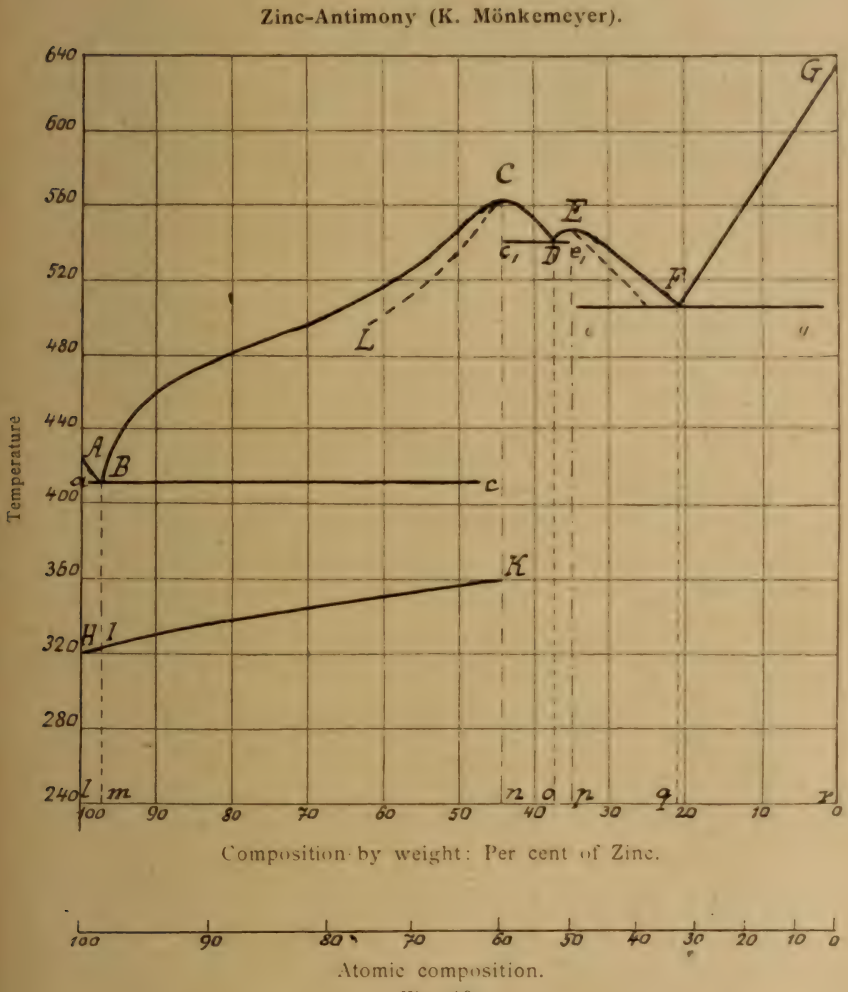
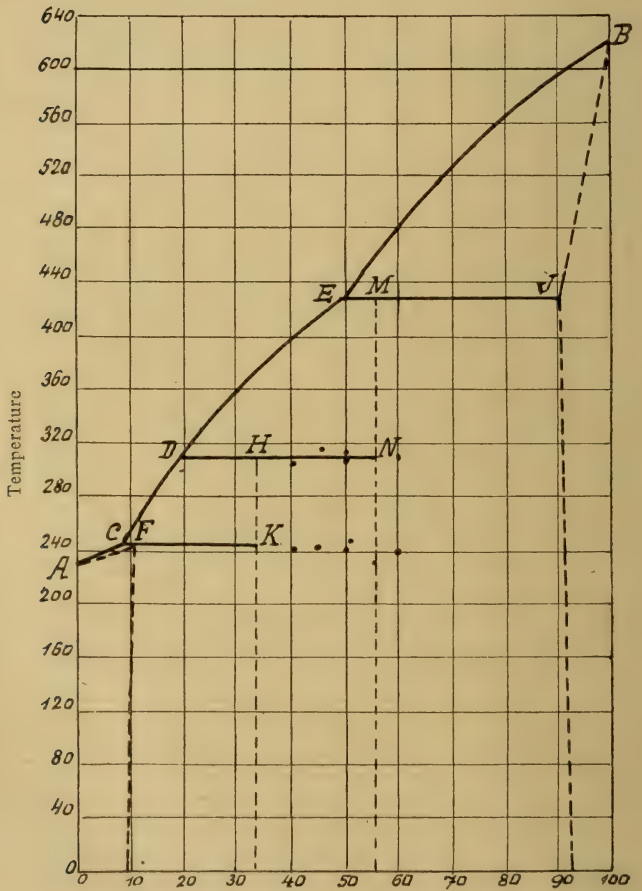


Fig. 10.

On account of the inconsistency which exists between the works of Messrs. Roland-Gosselin, Charpy and Stead with regard to the alloys of tin and antimony, Mr. Reinders has deemed it expedient to construct the diagram of equilibrium and to fix its regions (Fig. 11).

The first additions of antimony (up to 8 per cent), progressively raise the fusion-point of tin. It is, however, a solid solution which is separated. The rest of the diagram is formed of three branches *C D*, *D E*, *E B* and three horizontal lines *C F K*, *D H N*, *E M J*. The author does not form any decided conclusions. In order to interpret his curves with certainty, it is necessary to know the times of arrest on the eutectic lines. For this purpose one should apply Tammann's principle.

Tin-Antimony (W. Reinders).



Atomic composition: Per cent of Antimony.

Fig. 11.

M. The quenching of the alloys of lead and antimony. Dubox, meeting of French and Belgian members of the International Association for Testing Materials, on the 25th of March 1905.

The alloys of lead and antimony containing from 2 to 7 per cent of antimony show phenomena analogous to those observed with steels. When heating them between 180 and 240 degrees, their molecular state is constantly modified and this state may be fixed by sudden cooling, which results in a modification of the physical properties of the alloy, viz: the resistance to compression is very much increased.

N. Gold-lead. R. Vogel, Zeitsch. anorg. Chemie, XLV, 11, 1905.

This group of alloys affords an excellent application of Prof. Tammann's new method of determining definite compounds.

Pure gold is deposited along $A\,B$ (Fig. 12). From B starts a horizontal line $B\,b$, and the corresponding liberations of heat become a maximum at point B', indicating a definite compound which in this case is Au_2Pb. Along $B\,b$ the following reaction, expressed in gramme molecules, takes place:

$$0.722\ Au + \text{Liq}\ (1.278\ Au + 1000\ Pb) \longleftrightarrow Au_2Pb.$$

Likewise, along the horizontal $C\,c$, the liquid reacts on Au_2Pb resulting in the new compound $AuPb$:

$$0.625\ Au_2Pb + \text{Liq}\ (0.375\ Au + 1.375\ Pb) \longleftrightarrow AuPb_2$$

$d\,D\,d'$ is an ordinary eutectic line along which $AuPb_2$ and Pb are simultaneously deposited.

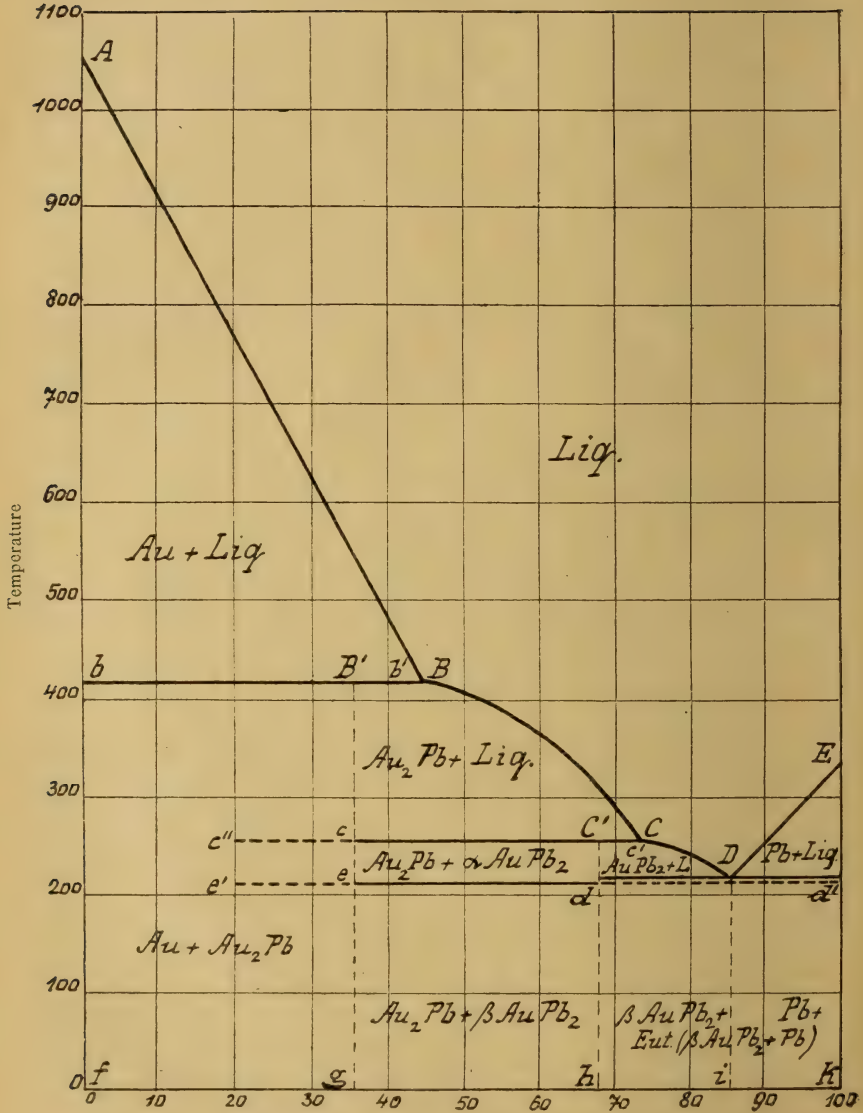
Finally, the horizontal e which almost coincides with $d\,D\,d'$, corresponds to a transformation of $AuPb_2$ in the solid state.

The reaction along $B\,b$ is very slow. At a little higher speed of cooling, gold remains behind which does not enter into reaction; the liquid is therefore much richer in lead and the lines $C\,c$, e are prolonged to cc'' and ee' respectively; i. e. beyond their theoretical region.

O. Gold-tin. R. Vogel, Zeitsch. anorg. Chemie, XLVI, 60, 1905. This case is analogous to the preceding one.

Mixed crystals are deposited along $A\,B$, point b indicating saturation. (Fig. 13.)

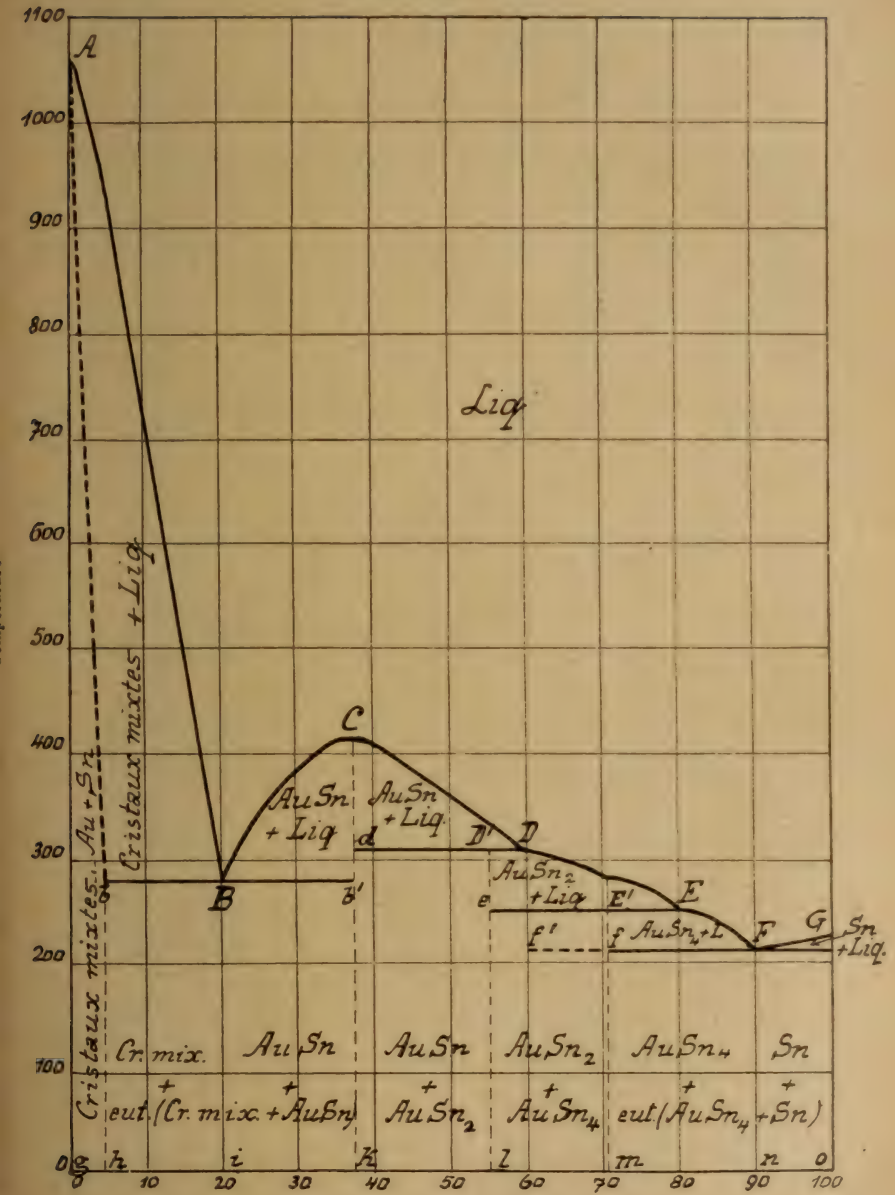
Gold-Lead (R. Vogel).



Composition by weight: Per cent of Lead.

Fig. 12.

Gold-Tin (R. Vogel).



Composition by weight: Per cent of Tin.

Fig. 13.

Cristaux mixtes = mixed crystals.

The maximum C immediately reveals the compound $AuSn$. The other compounds, to the number of two, do not appear on the liquidus-curve. It will be necessary to search for them, always applying the Tammann-method, on the horizontals.

The following reaction takes place along $D\,d$:

$$0.597\ AuSn + \text{Liq}\ (0.403\ Au + 1.403\ Sn) \rightleftarrows AuSn_2,$$

and along $E\,e$:

$$0.850\ AuSn_2 + \text{Liq}\ (0.150\ Au + 3.150\ Sn) \rightleftarrows AuSn_4.$$

The compounds $AuSn_2$ and $AuSn_4$ correspond to points D' and E' respectively.

The remainder presents no difficulties.

P. Silver-aluminium. G. I. Petrenka, Zeitsch. anorg. Chemie, XLVI, 49, 1905.

These experiments complete those of Mr. Gautier[1]; but the compound Al_3Ag_4 described by Mr. Guillet[2] is not found again.

Silver can keep up to 4 per cent of aluminium in solid solution, and forms with the latter metal two definite compounds $AlAg_2$ and $AlAg_3$. Each of these compounds can exist in two isomeric states.

Crystals of silver holding aluminium in solution, begin to be deposited along $E\,D$; following $E\,d$, the solidification terminates and d is the point of saturation.

On the horizontal $D\,d$ saturated mixed crystals are first deposited, then the compound $AlAg_3$ (point D).

Between D and C, we have mixed crystals of $AlAg_3$ and of $AlAg_2$ whose solidification begins as we follow $D\,h\,C$ and finishes following $D\,i\,C$.

Point C corresponds to $AlAg_2$ and it is this body which is deposited in following $C\,B$. $A\,b$ defines the deposit of aluminium and $a\,b$ is a eutectic line of Al and $AlAg_2$.

$AlAg_2$ is transformed along $F\,D'$, and $AlAg_3$ along the same line into mixed crystals which it forms with $AlAg_2$ and when it is pure along the straight line $D'\,d'$.

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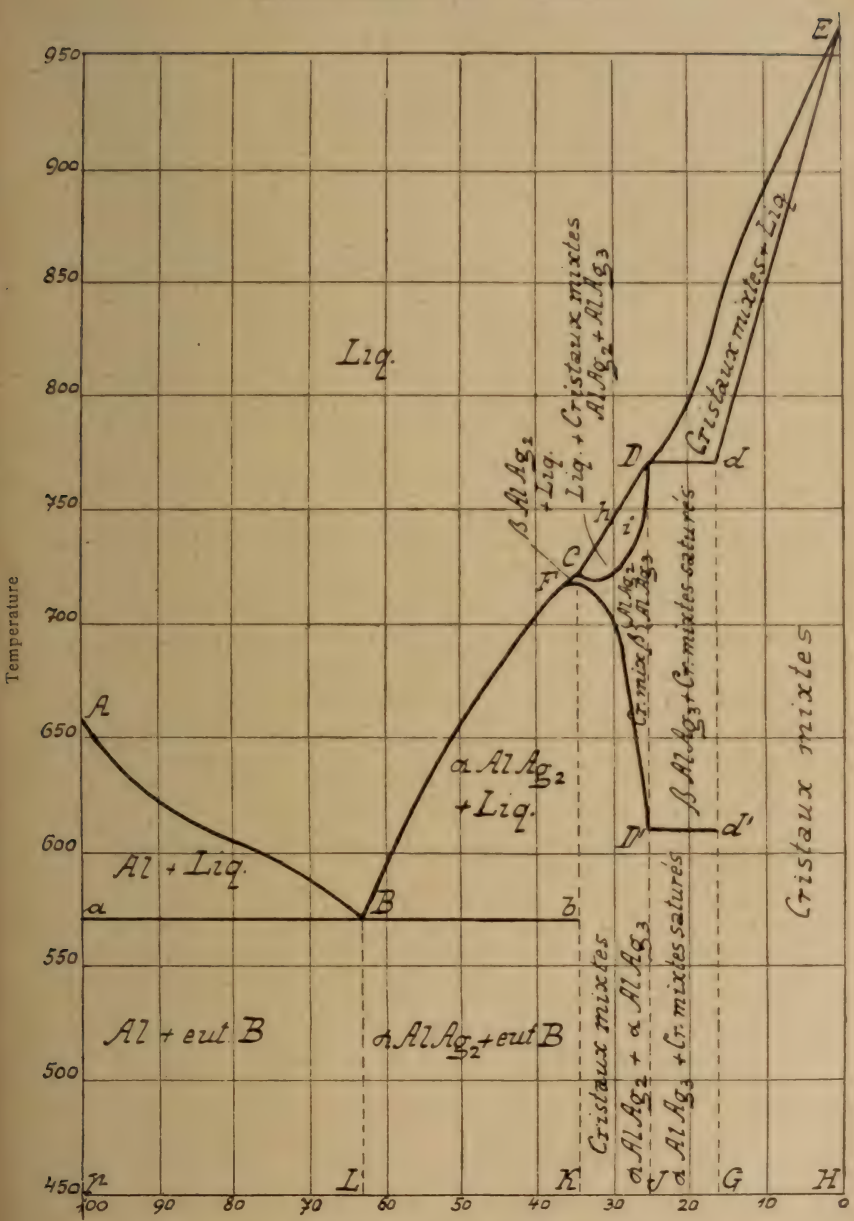
[1] Bull. Soc. Encour. (5), I, 1293, Oct. 1896.

[2] Thèse, Renouard, Paris, 1902.

Silver-Aluminium (G. I. Petrenko).



Atomic composition: Per cent of Aluminium.

Fig. 14.

Cristaux mixtes = mixed crystals.

Cristaux saturés = saturated crystals.

Q. Magnesium-thallium. G. Grube, Zeitsch. anorg. Chemie, XLVI, 76, 1905.

The curve (fig. 15) points out three compounds Tl_3Mg_8, $TlMg_2$, Tl_2Mg_3, which correspond to maximum C and to points D_1 and E_1 respectively and situated on the horizontal lines $D\,c$, $E\,o$.

The magnesium can hold up to 7 per cent of thallium, $a\,b$ and $g\,h$ are ordinary eutectic curves.

R. Copper and arsenic. A. H. Hiorns, Electro-Chemist and Metallurgist, III, 648.

The author has only determined the liquidus-curve, and that only partially. The curve is reproduced in fig. 16 and shows three maxima, which should correspond to the definite compounds Cu_3As, Cu_5As_2, Cu_2As.

These conclusions, which are given with due reserve, can only be considered as expressing a first approximation. It is evidently necessary to determine the solidus-curve and increase the micrographic tests in certain regions.

S. Copper and aluminium. These alloys have given rise to several researches.

The fusibility-curve has been traced by Mr. Le Verrier[1] and also by Mr. Le Chatelier[2]. It shows two maxima corresponding to the two definite compounds $AlCu_3$ and Al_2Cu. By means of micrographic researches, Mr. Le Chatelier[3] has subsequently been induced to admit of a third compound approaching the formula $AlCu$. Mr. Guillet[4] has re-discovered these three compounds by pure chemical methods.

Mr. W. Campbell has again[5] taken up the study of this subject. According to him, the alloys containing from zero to 54 per cent of copper, form a simple series with eutectic point towards 32 per cent of Cu. From zero to 32, it is aluminium; from 32 to 54, the compound Al_2Cu, which are deposited as we

[1] Lejeal, L'Aluminium, p. 168. (D'après Le Chatelier.)

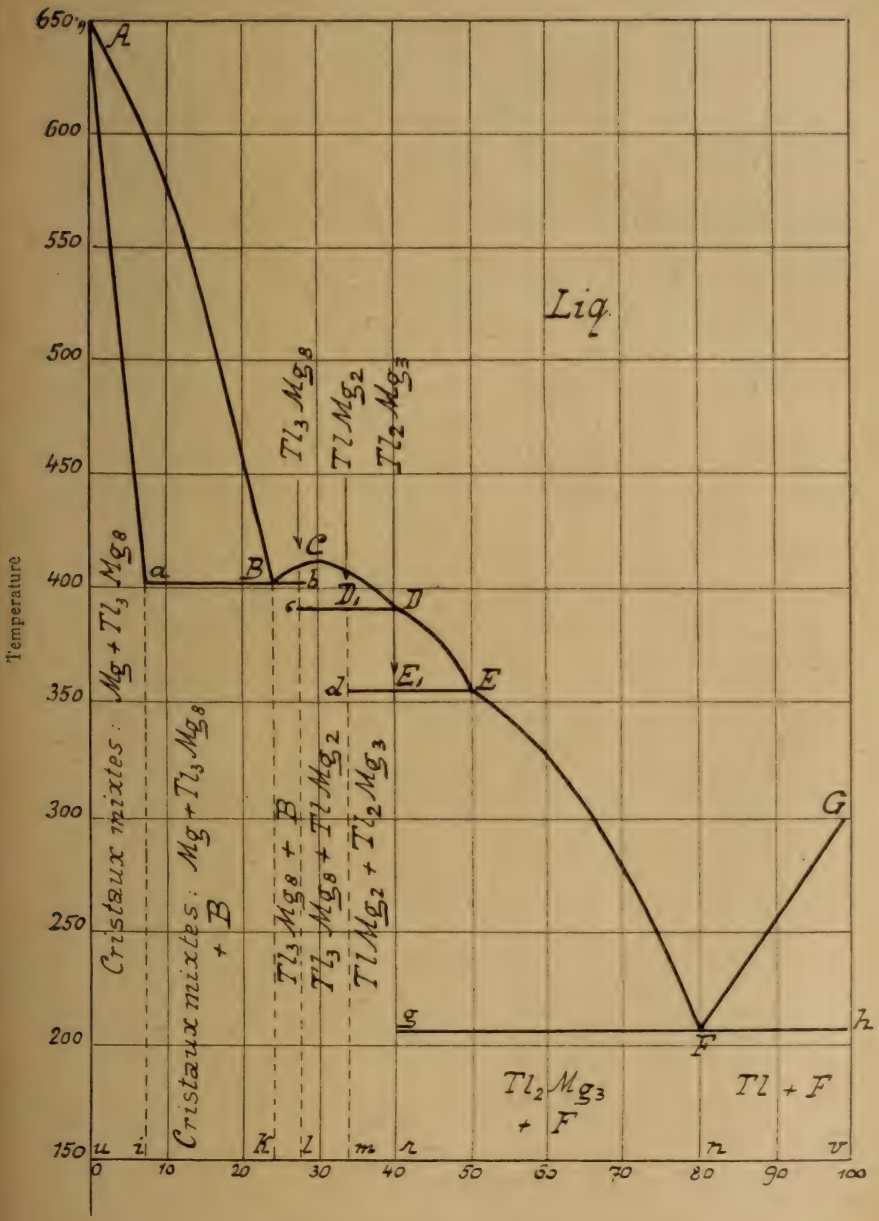
[2] Bull. Soc. Encour., (4), X, 574, May 1895.

[3] Bull. Soc. Encour., (5), IV, 380, Sept. 1900.

[4] Thèse, Renouard, Paris, 1902.

[5] Journ. Franklin Instit.; CLVIII, 161 (1904); also Revue de Métallurgie, I, 727, Amer.-chem. Soc., XXVI, fasc. 10, 1904.

Magnesium-Thallium (G. Grube).



Atomic composition: Per cent of Thallium.

Fig. 15.

Cristaux mixtes = mixed crystals.

follow the liquidus; but the crystals are never pure and contain about 2 per cent of copper or aluminium in solid solution.

Above 54 per ceut of *Cu*, Al_2Cu decreases and a new constituent appears which exists alone in the alloys with between 78 and 83 per cent of *Cu*. This compound cannot be identified because it always forms solid solutions.

From 83 to 90 per cent of *Cu*, the alloys solidify as solid solutions and resolve during slow cooling into a complex of phases to the left and right of a eutectic point situated at 87 per cent. This is what takes place during the quenching of these alloys; they thus show the structure of martensite.

Only solid solutions exist in the alloys with above 92 per cent of *Cu*.

Mr. Breuil has confirmed the facts with regard to quenching of an aluminium bronze, the exact composition of which is not given, and placed the transformation at between 670 and 730 degrees[1].

Finally, Mr. Guillet[2] has lately undertaken to trace the complete diagram. (Fig. 17.) By also examining the microstructure, he was able to distinguish 7 constituents:

α) containing from zero to 8 per cent of aluminium in solid solution;

β) which should be the compound Cu_3Al or a solid solution;

γ) solid solution which can only be preserved by quenching and which contains from 8·6 to 15·5 of aluminium;

δ) solid solution whose percentage of aluminium varies between 15·5 and 30·00;

ε) which appears to be the compound *AlCu*;

ζ) which is the compound Al_2Cu;

η) which is pure aluminium or a solution of aluminium-copper with a very small percentage of copper.

In summing up, these results generally confirm those which have already been obtained. It has not always been possible to record the liberations of heat, which indicate the end of solidification, notably between point *D* on the curve and the composition *AlCu*.

There are still some unsettled points.

[1] Comptes-rendus Ac. Sc., CXL, 587 (1905); also Revue de Métallurgie II, Extr., 429, August 1905.

[2] Étude théorique et industrielle des alliages de cuivre et d'aluminium, Revue de Métallurgie, Mém., II, 567, August 1905.

T. Copper and zinc. E. S. Shepherd, Journ. Physical Chemistry, VIII, 421, 1904.

Mr. Charpy[1] has already traced the fusibility curve and has found it to consist of two branches, intersecting in the neighbourhood of the point which corresponds to the composition $CuZn_2$; he considers this fact as a favourable argument for the existence of a definite compound of this formula, a compound which earlier research, conducted by other methods, seemed to indicate. Robert

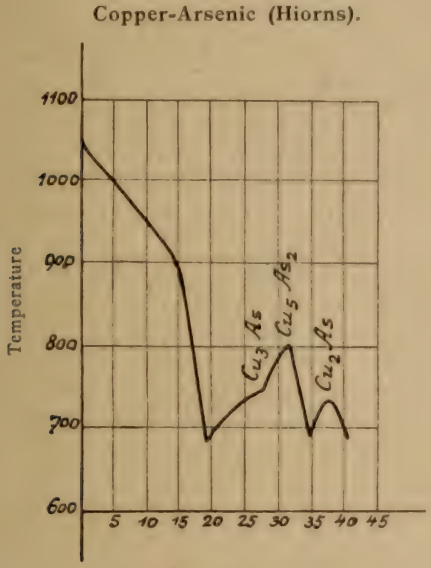


Fig. 16.

Austen and Mr. Stansfield have rectified and completed this first diagram; but the state of the science at that time, did not permit them to interpret their results with certainty[2].

The micrographic study has been entered upon by Messrs. Guillet[3], Behrens[4] and Charpy.

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[1] Bull. Soc. Encour., (5), I, 180, Febr. 1896.

[2] Fourth Report to the Alloys Research Committee, Proc. Mech. Eng., (fior. 1897).

[3] Comm. Meth. d'essai, I. session, Rapport particulier, II, 19 (Rapport du 5 avril 1893).

[4] Das mikroskopische Gefüge der Metalle und Legierungen, Voss. Leipzig 1894.

Behrens seems to infer the existence of two definite compounds $CuZn_2$ and $CuZn$, but Mr. Charpy, under due reserve, infers the existence of $CuZn_2$ and Cu_2Zn.

Mr. Shepherd does not think that any of these compounds exist. According to him, copper and zinc only form solid solutions, belonging to six different types, of which the respective regions are completely fixed in the diagram, fig. 18, viz:

α) 100 to 71 per cent of copper
β) 64 „ 45 „ „ „ „
γ) 40 „ 31 „ „ „ „
δ) 30 „ 23 „ „ „ „
ε) 19 „ 13 „ „ „ „
η) 2·5 „ — „ „ „ „

δ) disappears during cooling.

The alloys from 100 to 70 of *Cu* show, after cooling, crystallites similar to those of bronze, simply because equilibrium has not had time to be established; sufficiently prolonged annealing renders the structure homogeneous on account of the complete diffusion of zinc.

Between 30 and 20 per cent of *Cu*, transformations in the solid state take place: the corresponding alloys differ in structure according to whether they have been quenched or annealed.

U. Special brasses. L. Guillet, Revue de Métallurgie, II, Mém., 97 (Febr. 1905).

Mr. Guillet has examined the brasses containing lead, tin, aluminium, manganese.

The lead does not alloy: it facilitates turning, but lowers the mechanical qualities and should only be used for small pieces which are not much exposed to fatigue.

The other substances act at the same time as reducing agents and influence the properties in proportion to the quantity remaining in the alloy.

Mr. Guillet maintains the second of these points of view. The most important result of his study is the metallurgical equivalence of certain substances such as zinc, manganese and aluminium, which substitute each other in a solid solution, according to determined proportions, but different to the chemical equivalents. As is already well known when substituting nickel,

manganese and carbon for each other in steels, there seems to exist a fairly general law, theoretically and practically interesting.

It may be remarked that the mechanical properties may be tolerably different, though the structures are alike, according to the nature of the components of the solid solution. A solid solution

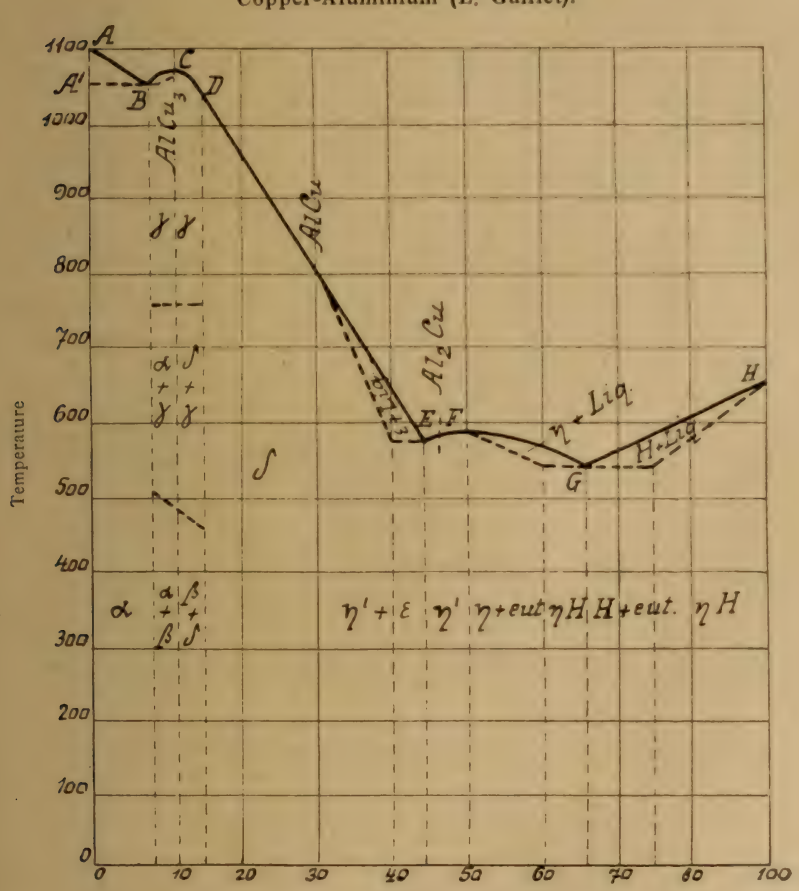


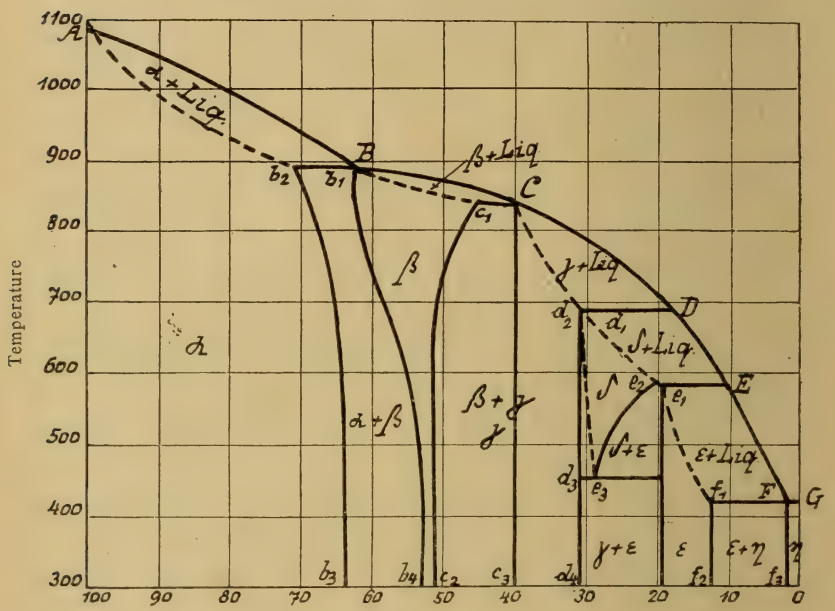
Fig. 17.

is not, like a definite compound, something unchangeable, to be either taken or left, but rather a substance admitting of all kinds of varieties, susceptible of more or less perfect adaptation for definite requirements.

V. Nickel and cobalt. W. Gürtler and G. Tammann, Zeitschr. anorg. Chem., XLII, 353, 1904.

Messrs. Gürtler & Tammann have determined the points of transformation of these alloys and also their points of solidification, which is a difficult task and has hitherto been given up by reason of the high temperatures to be measured.

Copper-Zinc (E. S. Shepherd).



Composition by weight: Per cent of Copper.

Fig. 18.

The result of these experiments shows that nickel and cobalt mix together isomorphically in all proportions, in their two states respectively. (Fig. 19).

X. Iron and manganese. Messrs. Levin and G. Tammann, Zeitschr. anorg. Chem., XLVII, 136, 1905.

The two metals form an uninterrupted series of mixed crystals. The ferro-magnetism remains until about 20 per cent of manganese is reached. (Fig. 19).

Y. Nickel and iron, cobalt and iron. W. Gürtler and G. Tammann, Zeitschr. anorg. Chem., XLV, 205, 1905.

The determination of the transformation points confirm practically earlier records. The fusibility-curve (fig. 19) seems to show a small maximum toward 35 per cent of nickel.

The relations between iron and cobalt are more complicated. The fusibility-curve, which is horizontal the greater part of its length, has a bend at about 8 per cent of cobalt.

Cobalt-Nickel (Gürtler and Tammann).

Iron-Mangan (Levin and Tammann).

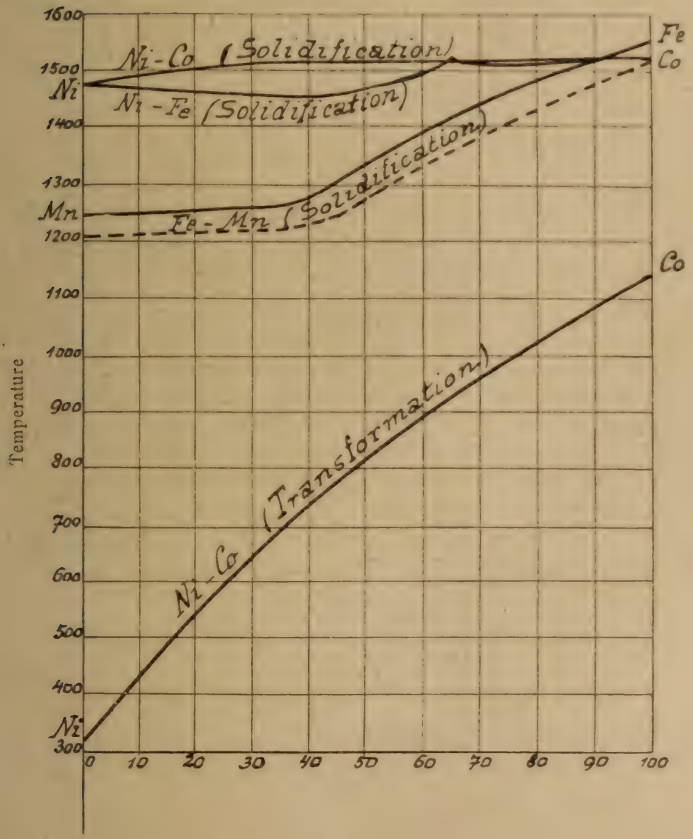
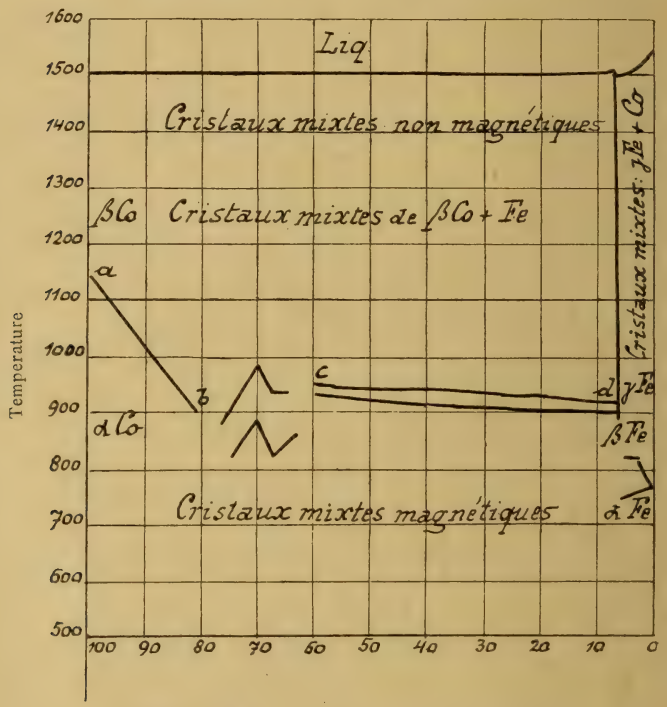


Fig. 19.

The curve of transformation (fig. 20) shows no less than four series of mixed crystals, viz: between 100 and 75, 75 and 60, 60 and 5, 5 and zero of cobalt.

The alloys of the first series are reversible. In the second series, the transformation is progressive within an interval of about 100 degrees with heating as well as with cooling. In the third series, a retardation takes place while cooling from 20 to 30 degrees.

Iron-Cobalt (Gürtler und Tammann).



Composition by weight: Per cent of Cobalt.

Fig. 20.

Cristaux mixtes = mixed crystals. Magnétique = magnetic.

There might exist between iron and cobalt a definite compound, mixable only in limited proportions with each of these metals.

Z. Iron and phosphorus. J. E. Stead, Journ. Roy. Microscopical Soc., p. 284 (1905).

Having previously studied the general relations between iron and phosphorus, Mr. Stead has now taken up a point of detail of

great practical interest, viz: the irregular distribution of the dissolved phosphorus. These irregularities seem to be related to the progress of solidification and perhaps to that of changes during heating. They react vigorously on the distribution of carbon.

Mr. Stead reveals them by means of a series of reactions, viz: by heat tinting a polished specimen and etching with a strongly diluted tincture of iodine, picric acid, etc.

AA. Iron and nitrogen. Hjalmar Braune, Rev. Métallurgie, II, 497 (1905).

This paper is followed by some interesting remarks from Mr. Le Chatelier.

The iron products, particularly in the presence of basic slags, by the intermedium of cyanides, seem to have the power of fixing some quantities of nitrogen, in reality very small as to absolute value, but which become very rapidly injurious and may lead to fragility.

The author has prepared some specimens containing different but known percentages of nitrogen, by heating the metal in an atmosphere of ammonia. As the quantity of nitrogen increased, the size of the grain diminished. At a sufficiently high percentage, the Neumann lamellae appeared which are a well-known indication of fragility. Iron containing nitrogen seems more sensitive to cold-hammering than ordinary iron: the striations from polishing, apparently effaced, reappear very easily by the slightest attack on the grains of a certain crystalline orientation.

It would be advisable to compare this study with that of Mr. Lecarme on cementation.[1]

According to this author, the superficial cementation with carbon is accompanied by a chemical transformation in the centre of the metal (a transformation which is probably due to nitrogen and produces fragility), which is generally attributed to the concomitant heat treatment.

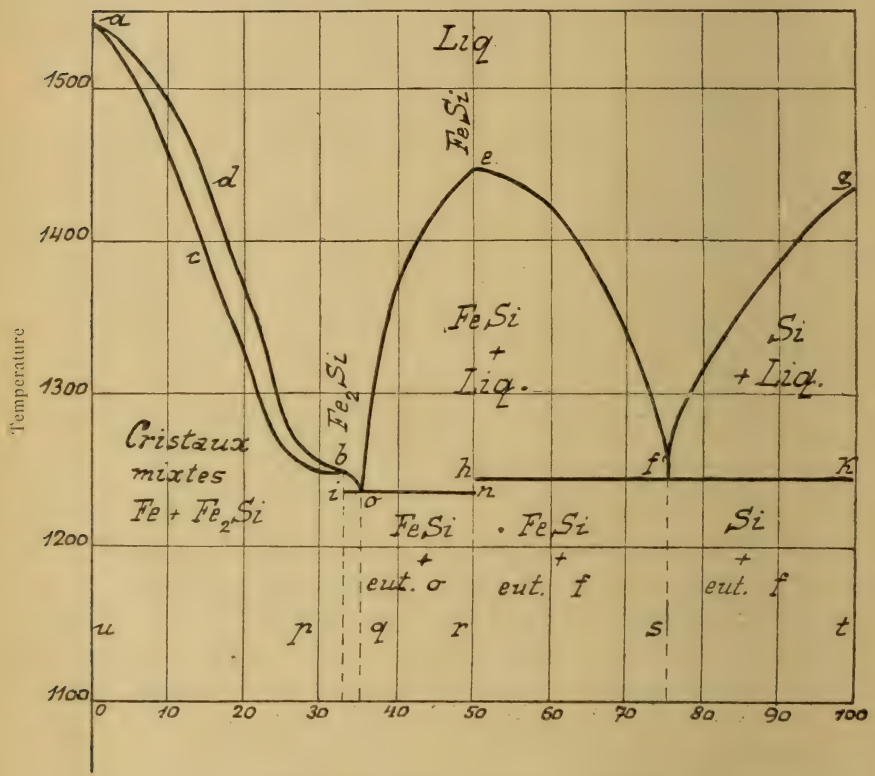
AB. Iron and scoriae. J. E. Stead, Iron and Steel Mag., IX., 105, Febr. 1905.

[1] Sur la fragilité des aciers doux cémentés. Rev. Métallurgie, II. Mém., 516 (July 1905).

It is well known that when turning large steel forgings, lines, visible to the naked eye, are often noticed, which English metallurgists denote by the name[1] of „ghosts“.

These lines are bands of ferrite, in the axes of which is found a trail of a mixture of sulphide and silicate of manganese. These

Iron-Silicium (Gürtler and Tammann).



Atomic composition: Per cent of Silicium.

Fig. 21.

Cristaux mixtes = mixed crystals.

substances are products of the first consolidation which the axes of the crystallites trace in the ingot.

Mr. Stead has carefully described the micrographic characters which permit of identification, and has determined their composition, at least approximately for the silicate.

[1] See: Greenish markings in the fractured surfaces of test-pieces, by H. C. Haworth, Journ. Iron and Steel Inst.: part II (1905).

AC. Iron & silicon. W. Gürtler and G. Tammann, Zeitschr. anorg. Chemie, XLVII, 163 (1905).

Messrs. Gürtler and Tammann have given a complete diagram of equilibrium for these two substances. (Fig. 21.) This diagram will be very useful for metallurgy.

Iron & silicon form by direct fusion two definite compounds, Fe_2Si and $FeSi$, previously described by chemists.

$FeSi$ corresponds to a very marked maximum of the curve (point e) and Fe_2Si to a slightly marked maximum on one side only (point b).

There are two eutectics, hk and in. The latter does not extend to the left of point b: in this region silicon forms with iron a series of mixed crystals, when the percentages in Si are lower than in Fe_2Si.

It is known that in the presence of carbon, Fe_2Si appears with much lower percentages in weight than 20. That is because carbon and silicon form a kind of alloy.

AD. Iron and carbon.

Roozeboom's diagram, reproduced in fig. 6 of the first part of this report, has been revised with great care by Messrs. H. C. H. Carpenter and B. F. E. Keeling [1]

As a result of this work, it seems that the lines SE and Ea run into one single continuous and practically straight line, Sa. If it really be so, then aBC is the line of the eutectic iron-cementite, the lines $E'B'$ and EF disappear, and finally the iron-graphite is no longer represented by anything, unless it be, in the alloys richer in carbon than 2·4 per cent, by some doubtful points of identification.

[1] Journ. Iron and Steel Inst., year 1904, part. I, 224. This was written when a paper on the equilibria of ferro-carbon by Mr. Charpy appeared (Comptes-rendus Ac. Sc., CXLI, 948, Dec. 4, 1905).

In this paper, Mr. Charpy, as a consequence of personal experiments, proposes to modify Prof. Bakhuis-Roozeboom's diagram according to the drawing fig. 22. The full lines refer to the equilibria of iron-graphite, and the dotted lines to the equilibria of iron-cementite. The eutectic aB (iron-graphite) is placed at 1150 degrees and the eutectic $A,B,$ about 15 or 20 degrees lower. According to the conditions of solidification, particularly according to the speed of cooling, the one or the other mixture is obtained, but not the two successively. Naturally the iron does not solidify in the state of pure iron, but of mixed crystals saturated with carbon, i. e. austenite.

For the present, we remain without any precise data regarding the equilibria between iron and graphite. It is no less certain that these equilibria constantly intervene in the operations of metallurgy; but they are probably generally affected by prolonged retardations, often even infinite, besides varying with the chemical composition and other circumstances more or less obscure (R).

Iron-Carbon (G. Charpy).

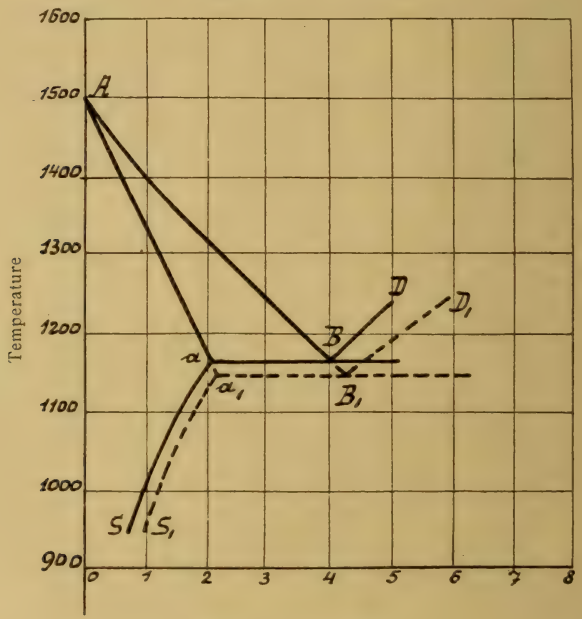


Fig. 22.

With regard to foundry iron, nothing orginal has appeared for the last two years; it seems that metallography is about to be introduced into foundries, especially in America, with a practical object, viz: to control the manufacture.

Mr. Alb. Sauveur,[1] with his well-known ability, has collected in a series of articles the prescriptions which are necessary for this speciality.

With regard to carbon steels, several researches may be quoted.

[1] Iron and Steel Mag., IX., 547 and X, 29, 309 and 413 (1905).

The question which remains the order of the day is that of structural constituents of steel. This subject has produced numerous objections, some futile, others of a personal character, and some interesting.

. There are no difficulties with annealed steels. The work of Sorby, interpreted in favour of modern ideas, seem to be decisive.

It is only the quenched steels which give rise to discussion.

The author of this paper has distinguished four structural constituents, which he has denoted by austenite, martensite, troostite and sorbite.

Austenite is obtained by quenching hypereutectoid steels in a bath of iced water, commencing at a temperature of at least 1000 degrees; but one never obtains it alone, if the steel does not contain foreign substances other than carbon. If besides carbon, one adds nickel or manganese or both in sufficient proportions, it is easy, even without quenching, to get the whole metal into the state of nickel or manganese austenite. It is practically non-magnetic, relatively soft, and permits of being deeply scratched by a sewing-needle.

Martensite is produced by quenching all carbon steels just above the critical interval: it is more or less hard according to the percentage of carbon and clearly characterised under the microscope by its needlelike structure.

Troostite is obtained, at the same time as martensite, by quenching hypereutectoid steels during the critical interval, by a mild quenching of these steels, or by an incomplete tempering of martensite or austenite.

Sorbite is produced by cooling just rapid enough to prevent the visible separation of cementite into lamellae, or by a complete tempering of martensite or austenite until the total liberation of the latent heat of the quenching.

These definitions seem very clear. The difficulty is this, viz:

1^0 that except austenite, which is a phase, γ-iron, the intimate nature of the other constituents is not exactly known;

2^0 that the four mentioned constituents do not form settled individualities. They are very different in many respects, when one takes them under a conveniently chosen form; but in reality they pass by imperceptible degrees into each other. They are divisions, necessarily somewhat conventional, in a continuous series, something

like the words „town", „borough", „village", „hamlet". Similar expressions are not less useful, but even necessary, so that one may judge for oneself in first approximation and on the condition of not expecting more from them than they really mean. If one wishes to distinguish a town from a borough, in a doubtful case, one seeks for numerical data, which in this case will be the number of inhabitants, and one lays down a certain convention. It is the same in the case of the constituents of steel, and when their name only does not seem to be a sufficient definition, one must define them more strictly by a numerical co-efficient, for instance, hardness or fragility or others, and if one co-efficient only does not suffice, then by two co-efficients.

These co-efficients are not yet determined. This might be done by the International Commission which Mr. Le Chatelier has instituted, with the co-operation of Dr. Glazebrook[1] for studying subjects of great philosophical value.[2]

Meanwhile we might make a survey of what has been attempted in the same direction by some isolated searchers.

Prof. Heyn[3] has carried out by himself almost everything which seems to be the programme of the commission. He has repeated all the fundamental experiments and has generally verified their exactness, and has, as one might expect from so competent a micrographer, carried out some original researches.

Prof. Heyn has only met with difficulties in the region of the austenite; but these difficulties seem now to be removed.

Mr. Kourbatoff[4] has applied his interesting reagents, which have already been mentioned above, to the study of the constituents of quenched steel, and particularly in the study of the tempering of austenite and martensite. Unfortunately that which he considered to be austenite was a hard constituent i. e. not true austenite, but a transition-form between austenite and martensite; the experiments are, however, to be repeated on authentic specimens.

[1] Projet de recherches sur les constituants des aciers. Revue Métallurgie, II, Mém., 329 (May 1905.)

[2] De la classification en chimie et de son application aux constituants des aciers. Revue Métallurgie, I. Mém., 207 (1904.)

[3] Bericht über die mikroskopische Untersuchung der vom Sonderausschusse für Eisenlegierungen des Vereines zur Beförderung des Gewerbefleißes hergestellten Legierungen. Leonard Simon, Berlin. — The Constitution of Iron-Carbon Alloys. Iron and Steel Mag., IX, 407 and 510 (May and June 1905).

[4] Rev. Métallurgie, 33, Mém., 169 (March 1905).

Mr. Kourbatoff has also been engaged with a constituent which has been mentioned in the first part of this paper (p. 261, with regard to Prof. Stansfield's work and which he calls troosto-sorbite. This constituent is obtained side by side with austenite and martensite, by quenching hypereutectoid steels from a very high temperature. It takes the form of specks, easily etched, is-localised along the joints, and in the interior of which lamellae of cementite have been isolated. It is quite true that the micro-graphic reactions are those of troostite, but on account of the manner in which the preparation has been made it would perhaps be more prudent not to affirm an identity which is not sufficiently established. It is for this reason that Mr. Osmond has never spoken of this constituent, although he had repeatedly seen it when making his researches on austenite.

Troostite has been the object of several researches. Mr. H. C. Boynton[1] has tried to show that this constituent does not contain carbon and that it is β-iron preserved by quenching. If this were so, β-iron might be preserved by quenching soft iron, which is not the case. Besides, in order to support this theory, one would have to limit the presence of troostite to hypereutectoid steels, i. e. to modify its definition, and that does not seem to be sufficiently justified according to facts. The experimental portion of this work is none the less interesting as a contribution to the study of difficult questions.

Mr. F. Rogers, on the contrary, is led to support the definition as mentioned above.[2]

Mr. C. Benedicks[3] advances a theory on this subject, which, although perfectly reconcilable with this definition, is new and very tempting: he assimilates troostite to colloids. This is a new point of contact between physical chemistry and metallography and is in any case a direction to be followed.

The same savant, in a very remarkable thesis,[4] has tried to show the existence of a new constituent of steels, „ferronite", which should be a magnetic form of β-iron. This conclusion is not without objections, but it is certainly brilliantly maintained by its

[1] Journ. Iron and Steel Inst., year 1904, part I, 262.

[2] Journ. Iron and Steel Inst., year 1905, part I, 484.

[3] Journ. Iron and Steel Inst., year 1905, part II.

[4] Landström, Upsala 1904.

author and supported by a quantity of well adopted experiments, in which all the resources of chemistry, physics and mechanics have been resorted to. It is a source abounding in records and ideas.

AE. Ternary steels.

Mr. Guillet has arduously pursued his monographs on special steels: nickel-, manganese-, silicum-, chromium-, tungsten-, molybdenum-, tin-, vanadium-, aluminium-, cobalt-, nickel-manganese- and nickel-vanadium-steels.[1]

The reporter will not dwell on these researches, as they ought to be the subject of a direct communication. They may, however, notwithstanding their abundance, be summed up in a few words.

In substance they confirm the law of metallurgical equivalences and have made it exact by numerical data. With the exception of some particulars of detail, one may say that whatever may be the substance added to iron, one never obtains, until the moment when the addition is sufficient to bring forth a definite compound, any other constituents than those of carbon-steels, viz: ferrite, cementite, sorbite, troostite, martensite and austenite.

It seems difficult to imagine a more striking demonstration of the theory which attributes the principal rôle in the variations of the derived irons to the variations in the iron itself. It is easy to find, in the new so-called rapid steels, such a composition as will give the whole gamut of constituents on a single specimen by progressive heating from ordinary temperature to glaring white, with or without subsequent quenching. Mr. Le Chatelier[2] has applied this experimental method: it is a very convenient application and prevents any transition from being lost.

Mr. H. C. H. Carpenter[3] has repeated by the heat method and by micrography, the study of carbon-steels with additions of chromium, tungsten or molybdenum. His experiments have been

[1] Bull. Soc. Encour., year 1903, 1er sem., p. 658 (Ni); 2e sem., p. 208 (Ni) et 421 (Mn). — Rev. Métallurgie, Mém. I, p. 46 (Si), 90 (Mn), 156 (Cr), 263 (W), 390 (Mo), 500 (Sn), 506 (Ti), 525 (Va); II, p. 312 (Al), 348 (Co), 350 (Résumé), 825 (Ni-Mn), 870 (Ni-Va).

[2] Les aciers rapides à outils, Rev. Métall., I, Mém., 334 (1904).

[3] The Types of structure and the Critical Ranges on Heating and Cooling of High Speed Tool Steels under Varying Thermal Treatment. Journ. Iron and Steel Inst. year 1905, part I, 433.

carried out with great care; they confirm what was already known: Tungsten and molybdenum, even during slow cooling, widen the critical interval and bring the lower termination right down into the region where the phenomenon stops. But to produce this effect, it is necessary for the cooling to commence at a sufficiently high temperature. The structure is always related to one of the types obtainable with iron and carbon alone, as we have already seen in the researches of Mr. Guillet. All these facts had besides been explained beforehand.[1]

The rôle of the chromium, according to Mr. Carpenter, is different to that of tungsten and molybdenum, which is not in agreement with the already described facts. But this divergence seems simply to be that the desired conditions were not realised.

The same savant together with Messrs. Hadfield and Longmuir has presented the seventh report to the Alloys Committee of the Institution of Mechanical Engineers. The subject dealt with in that report is the alloys of iron with nickel and carbon. It includes an extensive metallographic portion. The alloys chosen are intermediaries between Mr. Guillet's two series: the results might thus have been predicted by interpolation with tolerable accuracy.

III.

Modifications of an isolated phase or of a complex of phases in the extent of their regions. Thermic and mechanical treatment.

A. Structure and modifications of isolated phases in the interior of their region.

This chapter contains the applications of metallography to mechanics, particularly the study of the deformation of metals.

Messrs. Ewing, Rosenhain and Humphrey have worked at this subject since 1899, and have arrived at a theory of deformation founded on the sliding of the material parallel to certain reticular planes. These reticular planes are called translation-planes (gliding planes): they have been discovered in the natural minerals by the German crystallographer, Prof. Mügge.

[1] Revue de Métallurgie, I, Mém. 348 (June 1904).

Messrs. Osmond, Frémont and Cartaud in a paper, a résumé of which was to have been presented to the St. Petersburg Congress, accepting fully the results of Prof. Ewing and his associates, have expressed as their opinion that these crystalline deformations are not the only ones which take place, and that in the crystallised substances, particularly in iron, there exists another kind of deformation which may be qualified as amorphous, because it tends to destroy the crystalline equilibrium and to cause the material to take a form which approaches more or less to amorphism.

Mr. W. Rosenhain has responded to these objections.[1] His reply is very interesting; it supplies a number of new observations, particularly with regard to the joints of crystalline grains which are, to a certain extent, formed by a mixture of two adjacent crystals. Unfortunately it is impossible in such a general paper as the present one to give even an approximate idea of the arguments brought forward from both sides. It would be necessary to go into numerous details which would change the character of this communication. We must therefore refer to the original papers.

In another paper[2] Mr. Rosenhain describes a very ingenious process for determining exactly the forms of superficial deformations. This process consists in depositing on the surface (deformed after polishing) by electrolytic means a layer of another metal which takes the impression. A transversal cut is then made and the interesting part which is the surface of contact between the two metals, can then be polished without the edges being rounded, and photographed in all its purity under a high magnification.

Messrs. Osmond and Cartaud[3] have proposed a micrographic method for showing approximately the crystalline orientation.

It is easy to see the practical interest of these papers. Even if they only seem to elevate pure science, they aim at that which should be one of the greatest preoccupations of the Engineer, viz: the cause of accidental ruptures of metals in actual service and the means of obviating these accidents by conveniently directed tests.

[1] The Plastic Yielding of Iron and Steel. Journ. Iron and Steel Inst., year 1904, part I, 335.

[2] Further Observations on Slip-Bands. Proc. Roy. Soc., LXXIV, 557 (1905).

[3] Revue Métallurgie, II, Mém., 811 (Nov. 1905).

Thus the interval between theoretical and practical researches has speedily been crossed over.

When a metal breaks in service, there are never preliminary deformations, nor even concomitant deformations, except in connected pieces. This kind of rupture is with good reason attributed to dynamic forces and one has tried to imitate it by impact-tests on notched bars and by submitting other bars to alternating bendings according to Woeler's method.

What is the process of rupture in this case?

We owe to Messrs. A. E. Seaton and A. Jude[1] some interesting researches on the fracture of notched test-pieces by impact. The shock is regulated so that steels of good quality require a rather large number of blows before rupture and the specimen is turned back after each blow, whereby fracture is reached without notable deformation. One may thus conveniently study the progress of fracture by stopping the test when required before fracture is completed, and it has been found that in the case of mild steels the fracture is principally propagated across the ferrite by following the crystallographic planes, but rarely across the perlite and very seldom along the joints of the grains of ferrite. The alternating forces likewise fracture the test-bars across the ferrite and the ruptures in actual service present the same characters.

In the eutectoid steels rupture necessarily takes place across the perlite, which is the only constituent. It seems erratic and follows neither the joints of the grains nor the lamellae of the perlite.

Mr. F. Rogers[2] has also taken up this question of fatigue in iron and mild steel. He distinguishes two kinds of superficial lines: 1^0 in normal steels, a large number of short curved lines; 2^0 in overheated steels, scattered, straight, long lines. This distinction is exact; the latter lines are the only ones which foretell a dangerous metal.

Annealing a polished specimen to tempering colours, permits of showing certain lines of fatigue. A metal which is too fatigued cannot be restored by annealing.

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[1] Impact Tests on the Wrought Steels of Commerce. Proc. Inst. Mech. Eng. (1904).

[2] Loc. cit.

Mr. Sidney A. Houghton has searched in the same direction for the causes of a boiler-accident. His observations with regard to the localisation of the forces, rupture in consequence of the superficial layer of oxide, the corrosion of the metal following these lines and the production of internal crystalline deformations in the phosphorus irons, are ingenious and justified.[1]

Messrs. Frémont and Osmond[2] have arrived at analogous conclusions by examining the furrows of corrosion in boilers. The strengthened portions, ends, stays, etc., localise the dynamical forces and break the oxide along Lüders lines. The metal oxidizes along these lines, a new force splits the formed oxide, exposes again the metallic surface, and the oxidation progresses according to the same mechanism, cutting slowly through the plate. These cuts are particularly dangerous when one has to do with fragile plates where fatigue has already set in on all sides of the lines of internal weakness. It is in this case only that explosions may be feared. In the good plates the furrows of corrosion only induce leakage of steam and one is thus warned by these leakages.

Mr. R. Job[3] enumerates the causes of failure of rails in service. They are, viz: 1^0 — pipes in the steel; 2^0 — presence of a considerable proportion of blow-holes; 3^0 — excessive segregation; 4^0 — coarse granular structure; 5^0 — rough handling.

It will thus be seen that the first four causes belong to metallography and Mr. Job has in fact made use of the microscope for his investigations.

Mr. Andrew[4] has continued his microscopic examinations of broken and deteriorated pieces.

B. Modifications of a complex of phases in its region. — There has not been any work done during the last two years which can be exactly classed under this heading.

[1] Note on the Failure of an Iron-Plate through Fatigue. Journ. Iron and Steel Inst., year 1905, part I, 383.

[2] Rev. Métallurgie, II, Mém. 773 (Oct. 1905).

[3] Some causes of Failure of Rails in Service. Iron and Steel Mag., X, 97, Aug. 1905.

[4] Wear of Steel Rails on Bridges. Iron and Steel Inst., year 1905, part II.

C. Indirect modification of a complex of phases.

Mr. P. Longmuir, who has previously studied the influence of the variations of the casting temperature on the mechanical properties of bronze, foundry iron and brass, has extended his researches to iron and steel-castings.[1] As in the case of the former metals and alloys, this influence of the casting temperature which seems to be characterised by the state of the joints, more or less survives all thermic and even mechanical treatments. It is indelible like congenital damage. The casting temperature must neither be too high nor too low. The author has tried to find out whether it records itself in the microstructure with recognizable characters, but he has not been able to come to any conclusive results. It is not at all certain that he has found the best means to arrive at his object. This question is important and deserves to be repeated. The initial distribution of impurities may play a role, and the processes, mentioned above, which Mr. Stead has applied in his researches on the distribution of phosphorus might be useful in this case also.

Messrs. A. Windsor Richards and J. E. Stead have continued their work on overheated steel.[2] This steel may be restored by annealing, and greatly improved by a treatment capable of transforming the perlite into sorbite, by quenching and tempering, double quenching etc. Comparative tests have been carried out by tension, bending, and by a method derived from that of Woeler. Tension-tests by themselves do not permit of judging a mild steel.

On the 15th of January 1904, Mr. W. Gowland presented the 6th report to the Alloys Committee of the Institution of Mechanical Engineers. This report is a study of the influence of various heat treatments, annealing and quenching, on the mechanical properties of a series of steels with all percentages of carbon. Microphotography has been applied to all the specimens after each treatment. Unfortunately this important work was interrupted by the regrettable death of Sir W. Roberts-Austen who conceived the idea and directed the work from the beginning.

[1] Journ. Iron and Steel Inst., year 1904, part I, 420.

[2] Journ. Iron and Steel Inst., year 1905, part II.

Messrs. R. Sankey and K. Smith[1] have studied some chrome-vanadium steels on a similar plan. They have rightly not limited their mechanical trials to tension-tests, but have also carried out hammer bending tests, impact-tests on notched bars and alternation-tests with Prof. Arnold's machine. The different methods may thus be compared, and it is a very instructive comparison which shows once more the insufficiency of tension-tests. The micrographic portion of the paper is extensive, but has been done under difficult conditions and has not given much new information.

IV.

Miscellaneous.

A. Copper, tin and oxygen. — E. Heyn and O. Bauer, Mitt. Kön. Materialprüfungsamt, Berlin 1904 — (also Rev. Métallurgie, II., extraits 364).

Prof. Heyn, who has previously studied the action of oxygen on cast copper, has extended his researches to bronze. He finds that oxygen unites with the tin and forms stannic acid, which renders the metal pasty, and may be eliminated by reducing agents such as phosphorus.

It is a happy application of metallography to an everyday commercial operation, about which as yet only little scientific data exist.

B. Study of alloys for shaft-bearings. — G. H. Clamer Journ. Franklin Instit. CLVI (also Rev. Métallurgie, I, Extr. 144).

Generally speaking all the alloys used in practice for bearings of a satisfactory quality contain at least two different structural elements, i. e. a hard element capable of supporting the desired load, and a soft element serving as a plastic support for the hard elements. The harder the surfaces of contact are, the smaller is the co-efficient of friction. As good working depends especially on a perfect fitting and as this perfection is neither realised nor above all maintained in service, the soft constituent is essential in order to produce some kind of automatic adjustment.

[1] Proc. Inst. Mech. Eng., read Dec. 1904.

The author points out the compositions recognized as the best and the corresponding structures.

C. Applications to Archaeology. — Every piece of metal carries its history inscribed in itself. Metallography, in sufficiently experienced hands, permits of obtaining interesting information on the primitive processes of metallurgy, and these processes are often much more skilful than one is tempted to suppose.

M. Chesneau[1] has examined some pre-historic bronze from Charente and Meuse. Arth and Lejeune[2] have studied a steel, likewise pre-historic, found in the neighbourhood of Nancy.

A cast bullet, found in the old pits of the Bastille, has been studied by Mr. Porlier.[3] The cementite had remained intact, while the iron of the perlite had been oxidized right through. The bullet had, however, kept its shape.

Paris, 16th of December 1905.

[1] Comptes-rendus, Ac. Sc. CXXXVII, 653 (1903).
[2] Rev. Métallurgie, II, Mém., 789 (Oct. 1905).
[3] Ib., II, Mém., 792 (Oct. 1905).

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INTERNATIONAL ASSOCIATION FOR TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 7.

ON THE RELATION OF CHEMICAL COMPOSITION TO THE WEATHERING QUALITIES OF BUILDING STONES; THE INFLUENCE OF SMOKE ESPECIALLY SULPHUROUS ACID ON BUILDING STONES; THE WEATHERING QUALITIES OF ROOFING SLATES.

REPORT BY THE CHAIRMAN OF COMMISSION 7, A. HANISCH, BAURAT, K. K. PROFESSOR.

APPENDIX I: TESTS FOR RESISTANCE TO WEATHER OF SANDSTONES BY **PROFESSOR DR. H. SEIPP.** (TRANSLATED BY R. N. LUCAS.)

APPENDIX II: RESULTS FROM TRIALS ON SWISS ROOFING-SLATE, TOGETHER WITH SOME IMPORTATIONS.

A) THE FORMATION AND TEXTURE OF CLAY-SLATE, BY **PROF. DR. A. HEIM.**

B) RESULTS OF THE PHYSICAL-CHEMICAL RESEARCHES BY **PROFESSOR L. v. TETMAJER (†).** (TRANSLATED BY CH. CAPITO, LONDON.)

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Problem 7:

a) On the relation of chemical composition to the weathering qualities of building stones; the influence of smoke especially sulphurous acid on building stones.

b) The weathering qualities of roofing slates.

Report of the Chairman of Commission 7, k. k. Professor, Baurat A. Hanisch. 1

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Appendix II:

Results from Trials on Swiss Roofing-Slate, together with some Importations*)

a) The Formation and Texture of Clay-Slate, by Professor Dr. A. Heim 27

b) Results of the physical-chemical researches by Professor L. v. Tetmajer † 45

*) At the desire expressed in several quarters of the International Association, these papers, already published in the Communications of the Swiss Federal Institute for Testing Materials (Eidgenössische Materialprüfungsanstalt) at Zurich in 1900, have been reprinted here.

a) On the relation of chemical composition to the weathering qualities of building stones; the influence of smoke especially sulphurous acid on building stones; *b)* the weathering qualities of roofing slates.

(Taken over by the Zurich Congress 1895).

Report of the Chairman of Commission 7, k. k. Professor Baurat

A. Hanisch.

(Translated by R. N. Lucas.)

Members of the Committee appointed for solving the above problem:

Chairman:

Hanisch A., Baurat, k. k. Professor, Vorstand der Prüfungsanstalt für Bausteine an der k. k. Staatsgewerbeschule in Wien.

Vice-Chairman:

Larivière P., Ingénieur, Paris.

Members:

Germany:	Dietrich E., Professor a. d. techn. Hochschule, Berlin.
	Glinzer E., Dr., ordentl. Lehrer d. allg. Gewerbeschule und Baugewerkschule, Hamburg.
	Seipp H., Dr., Ingenieur und Direktor der kgl. Baugewerkschule in Kattowitz.
England:	Kennedy A. P. W., F. R. S., London.
	Siemens A., London.

Norway:	Vogt H. L., Professor der Universität Christiania.
Austria:	Greil A., Bauinspector, Vorstand d. städt. Materialprüfungsstation, Wien.
	Tetmajer L. v., Dr., k. k. Hofrat, o. ö. Professor der techn. Hochschule in Wien, Präsident d. Verbandes (†).
Russia:	Kournakoff N., Professeur, institut des ingénieurs des mines, St. Pétersbourg.
	Lamine N., Prof. adjoint à l'inst. des ing. des voies des comm., St. Pétersbourg.
	Maluga J. Professeur à l'académie des ing. milit., St. Pétersbourg.
	Perrimonde, Ingénieur, St. Pétersbourg.
Sweden:	Wikander A., Professor, Göteborg.
Switzerland:	Grubenmann, Dr., Prof. am eidg. Polytechnikum, Zürich.
	Lunge G., Dr., Prof. am eidg. Polytechnikum, Zürich.
Hungary:	Wartha V., Dr., Professor am kgl. Polytechnikum, Budapest.
United-States:	Kemp J. F., Geologe, Prof. a. d. Universität, New-York.
	Merrimann M., Ingenieur, Prof. a. d. Universität, South-Bethlehem, Pa.

Unfortunately death has robbed the Committee of one of its most active members, namely, Prof. Hans Hauenschild, Geologist and Chemist of Berlin. The deceased gentleman was one of the original founders of the International Association and had already about 1880 commenced discussions with Prof. Tetmajer in regard to the preservation of building stone, and the classification and sub-division of building material. As Chairman of the Committee on «Methods for examining into the quality and resistance to weather of slates», Prof. Hauenschild laid before the Congress of Zurich in 1895 a lengthy and elaborate report which has had the effect of largely contributing to the success of a work we have taken in hand. May his memory be ever held in honour!

Prof. Brunner of the University of Lausanne, overburdened as he was with work and a variety of distracting duties, also found it necessary to retire from the Committee, and the Committee was consequently reluctantly and sorrowfully compelled to abandon all hope of co-operation on his part.

At the end of 1902, the Chairman addressed a circular letter to the various members of the Committee in the following terms:

To the members of Committee 7.

The following problems are partially dealt with in the publications which appeared in 1900 in the new edition of the first volume of the communications of the Zürich Institute for Testing Materials, by Prof. Tetmajer, and the work dedicated to Committee No. 7 by Prof. Dr. Seipp on the resistance to weather of natural building stone with special reference to slates. The President of the Association enabled me to circulate these works before the meeting of the Budapest Congress with the request to subject them to a thorough examination, and to be good enough to forward your views on the questions *a* and *b* to the Committee.

To this circular letter I received only one reply, namely, from Prof. E. Dietrich which, ran as follows:

Berlin, 3rd January 1901.

My dear Sir,

After sending on the two works of Prof. Hauenschild I reply as follows:

Quest. *a* is more of a chemical than of an architectural nature, and

Quest. *b* is difficult to form an opinion about. All the same I have read the matter with interest, and I accordingly desire to place before the Committee the following questions:

1. Is it, as Martens of Berlin maintained to be the case for homogeneous masses, unimportant what size the test-pieces are? I am quite unable to believe this assertion.

2. The quarry owners naturally send samples from their best beds, but also employ the less valuable. What use then are tests? Should it not be insisted upon that when samples are sent they should be attested by a local authority as being taken from both the more valuable and less valuable beds?

3. Is it not of importance in the case of sedimentary rocks to take note of the stratification, and to indicate whether the pressure or tension has been applied parallel to or at right angles to stratification?

4. Is the parallel guiding by the two screws in the machine, as indicated on page 78 of Tetmajer's paper, so reliable that the upper guide plate can be dispensed with? Even if new screws are exactly alike they may wear down differently after a time.

5. In frost tests should not the assistance of the lens be made use of so as the better to observe any alterations on the surface of the material tested? Should not this be universally prescribed?

Yours sincerely

E. Dietrich.

Circular letter

to the members of Commission 7.

As I have been accorded the honour of again being selected as head of the Committee 7, I would be glad if its members would be good enough to express their views on the statements made by Prof. Dietrich. At the meeting of the Budapest Congress a communication was read by Councillor Eger on the testing of the building materials employed by the Prussian Ministry of Buildings, which gives further information concerning the researches of Professor Hirschwald on a basis for determining the resistance to weather of natural building stone. As Committee No. 7 expressed the desire to postpone their labours until the work of Prof. Hirschwald had appeared, the following ad interim report was drawn up. The material to be tested was taken from 1000 different sources, and where the origin of the

stone was known and the quarry from which it was obtained worked, the samples were taken from the freshly quarried materials. In treating the samples the following principles were in general observed.

1. The determination of the qualitative and quantative chemial composition of the stone involving both by rough analysis and special analysis. Where the composition of the weathered surface pointed to the conclusion that chemical decomposition had taken place, both the interior and weathered portions of the stone were analysed.

2. A determination of the mineral constituents, the particular development and structural relationships to the stone were determined by the microscopic examination of slides prepared from the specimens.

3. Examination of the size and arrangement of the cavities in the stone was also undertaken, and any interconnection between them noted.

4. The determination of the amount of water the stone absorbs at its ordinary pressure; the amount it absorbs in vacuo; and the amount it absorbs at a pressure of 100 atmospheres, for the purpose of determining the porosity co-efficient and the amount of water which can be got into the pores or interstices at ordinary pressure.

5. The determination of the so-called adherents of the stone, that is to say, the strength with which its constituents are cemented together.

6. The determination of the amount of softening of the stone produced by water.

7. The frost test by repeated freezing of the stone when saturated with water.

In requesting that this method of testing may be considered by the Committee, I would direct attention to the excellent work of Tetmajer and Seipp, and request that all studies and experiments made in regard to the above questions may be forwarded to me in order that we may approach the ultimate solution of the problem.

(Signed) **A. Hanisch,**
Chairman of Committee No. 7.

Vienna, December 1902.

The only answer I received to this circular letter, which was despatched in all directions and to all countries in the world, was the following:

9th February 1903.

Dear Sir,

Your circular letter of 1902 had now first reached me. It has greatly interested me, and I am pleased to learn that so many careful experiments are being conducted in regard to the subjects in question. There is the greater reason therefore that I should abstain from dealing with the subject, more particularly as I am greatly occupied with other work for the International Union.

The remarks of Prof. Dietrich appear to me absolutely to the point.

Respectfully yours
G. Lunge.

A second letter directed to the members of the Committee was as follows:

Vienna, May 2nd 1903.

Dear Sir,

The Council of the I. A. T. M. decided at its last sitting that the manuscripts of all reports and lectures for the St. Petersburg Congress of 1904 must be sent on to the President of the Association by the 15th of January 1904.

The undersigned therefore requests you to let him have the material a little before that date, say by the end of the year, so that the printing may be proceeded with.

Yours respectfully,

Prof. **Hanisch**,
Baurat,
Chairman of the Committee for Problem 7.

A letter from Dr. Lunge then came to hand making the following statements:

Dear Mr. Chairman,

I have received your letter of the 2nd of May, but must inform you that it will be scarcely possible for me to take part practically in the solution of problem 7, as all the time I have to spare for such questions is taken up by other Committees, of one of which I am chairman.

Yours respectfully,

Prof. Dr. **Lunge**.

It looked, therefore, in the spring of 1903, as if there were no chance of making a start with the solution of the problems. Then appeared Director Dr. H. Seipp (who had already put before the last Congress his important »The resistance to weather of the natural building stones, with special reference to roof slates,«) with an important work for the Association. This work forms a completion of a previous work and is of the same general excellence. (Appendix to Seipp's Tests for resistance to weather.)

Of very special importance for the final solution of problem 7 regarding the »Method of examining the quality, particularly the resistance to weather, of roof slates« is the following highly interesting work of Professors Dr. A. Heim and L. v. Tetmajer. (Appendix, page 27.)

In what follows certain experimental results recently obtained by the Chairman with ordinary slates are shortly given.

In submitting the above works to the Brussels Congress I beg to thank my colleagues for their assistance, and trust that we have succeeded in bringing the various questions somewhat nearer to a solution.

A. Hanisch,
Chairman of Committee, No. 7.

Vienna, January 1906.

Material (Roof slates)	Effervesces powders in hydrochloric acid	Specific gravity determined by hydrostatic balance	Water taken up in four weeks in weights per cent			Co-efficient of flexure in kg per cm²						Loss of weight in per cents of the original weight. Effect ascertained in per cents by boiling the slates in hydrochloric acid, 1 pt. to sulphuric acid 1 pt. Water 98 pts. by weight
						Dry			Wet			
			minimum	maximum	Mean	minimum	maximum	Mean	minimum	maximum	Mean	
English red	a little	2·85	0·09	0·11	0·10	743	804	771	656	800	715	0·07
English blue . . .	scarcely observable	2·83	0·23	0·24	0·23$_5$	560	729	653	500	672	574	0·33
French red	do.	2·84	0·19	0 25	0·22	500	888	674	325	459	395	0·15
French blue	do.	2·83	0·11	0 21	0·16	570	643	609	364	638	524	0·30
French green . . .	no	2·83	0·18	0·26	0·22	500	612	550	338	481	423	0·13
French blue sand to be from Johannes quarry	strongly	2·74	0·22	0·27	0·25	461	506	478	207	433	345	0·77
Moravia and Silesia	no	2·79	0·21	0·33	0·29	452	606	535	333	611	439	0·22
Rheinish	do.	2·77	0·21	0·34	0·28	486	576	539	269	345	323	0·12

Appendix I.

Tests for resistance to weather of sandstones.

By

Prof. Dr. H. Seipp,

Director of the Baugewerkschule at Kattowitz.

(Translated by R. N. Lucas.)

The following communications are intended to constitute a small contribution to the practical examination of the resistance to the weather possessed by natural building stones. The researches refer to the examination of a single group of building stones, namely the sandstones — a group the importance of which is considerable — and are restricted to a small number of interesting examples. Finally, these experiments test only the «artificial resistance to weather», the principles and methods employed in which have been described in my work «The resistance to weather and tests of resistance to weather of natural building stones with special reference to roofing slates» (Jena H., Costenobles 1900), in which the subject was first treated exhaustively, and which was submitted to the Budapest Congress in the autumn of 1901. As in those researches roofing slates formed the principal subject of experiment, on the present occasion the important group of sandstones, so much used for building purposes, is treated of. The kind of sandstone employed was as follows:

1. A sandstone of special interest on account of its «weathering», the origin of which was not precisely known, but which has been described as «sandstone from Essen». This stone was employed in 1848 for facing the south side of the Minster at Essen

on the Ruhr (Gothic extension of the Church). Prisms of this material have shown better marked traces of weathering. It is a fairly fine grain compact sandstone, light grey in colour. Under the lens are observable, in addition to the quartz grains and binding material, a dark coloured glittering constituent (schist and chlorid detritus together with hydrated ferric oxide) and, more sparingly, potash mica. The quarry from which this stone was obtained was probably Staudernheim on the Nahe.

2. Hils sandstone from the quarry of Herr F. Sotter (at Delligsen) not far from the railway station at Naensen, in the Grand Duchy of Brunswick. A very fine grained, dirty-white grey and occasionally spotted or striated sandstone. The grain was the finest of the series 1—9. Under the lens it shows in tolerably large quantity a black constituent (probably slate or schist detritus with chlorites and also ferric hydrate).

3. Solling sandstone, and particularly Stadtoldendorf sandstone, from the Stadtoldendorf quarry on the line between Holzminden and Kreiensen, Grand Duchy of Brunswick. The proprietor cannot be decided with certainty (well known firms being C. Reese & Co., Stadtoldendorf, E. Rothschild, Stadtoldendorf and Haarmann & Co., Holzminden). This is generally a light material much employed in the Weser district, and has a fine grained structure with some potash mica and a light red colour, with blue attrition surface.

4. Nörten sandstone from the quarry in Rodetal near Nörten (eastern spur of the Solling hills) belonging to Herr Franz Wenig, Nörten, near the railway from Carlshafen to Northeim. A fine red moderately fine-grained material (not so bright red as No. 3 and somewhat coarser, with rough small whitish spots from 2 to 5 mm in size and 1—10 cm apart due to calcarious inclusions). In addition there is a certain amount of potash mica present, and the binding material is also calcareous.

5. Hardegsen sandstone from the quarry of Herr Schonlau of Hardegsen at Hünschenburg (eastern spur of the Solling hills), station on the Carlshafen Northeim line. Colour almost exactly the same

as No 4 (without the bright spots); the size of grain, too, is much about the same, though perhaps slightly greater and the content of muscovite quite small (the original here says muscovitz but presumably muscovite is meant. Transl.).

6. Osterwald sandstone from the quarries of the firm Meine & Illemann at Osterwald on the line between Hameln and Elze, in the province Hanover. Fine, almost white, sometimes slightly yellow material, the grain being about the same as with 4 and 5.

7. Mehle sandstone from the quarries of the last named firm at Mehle on the Hameln and Elze line. Whitish-grey and otherwise similar to 6, though somewhat more fine-grained, the grain being about the same as in 4.

8. The Wrexen sandstone from Wrexen in Waldeck, quarry owner Herr C. Wöllbrinck in Wrexen, railway Scherfede. Light reddish yellow sandstone of the same sized grain as No. 5.

9. Obernkirchen sandstone (in Denmark called Bremen sandstone) from the quarries in the Bückeburg near Obernkirchen, not far from Bückeburg on the line Rinteln to Stadthagen. A fine white sandstone (less grey than 7) fine grained, somewhat like 1 or 7, almost free from mica with siliceous binding material (quartz sandstone).

Simultaneously with the above mentioned tests of sandstones further tests were made with:

10. a white sandstone from Regen in Bavaria, obtained from the office of Dr. Krantz of Bonn; pure white, very close milky-looking material with a few yellow spots here and there.

11. Odenwalde syenite, scientifically the diorite of Lindenfels in the Odenwald, in the Grand Duchy of Hessen; greenish-black stone with white spots and fairly fine grain crystalline structure, probably from the quarries of Kreuzer & Böhringer, Lindenfels.

12. Carrara marble (statuary marble), pure white, fine grain crystalline structure.

From the explanations given in my work stated above, pages 43 and 44 and 176 to 179, it was proved that the capacity of different stones to resist weather was best ascertained by two sets of tests, viz.

1. The frost or freezing test.
2. The test with reagents.

Both sets of tests, which only together provide a measure of capacity for resisting weather, have been carried out. Various other researches which should be included have not been introduced in the present report as they have not been concluded. In the case of 11 and 12 and also the «sandstone from Essen» the frost test was omitted, as this was employed for the second sort of Wrexen sandstone. This, howewer, has not been subjected to the other tests, and it is not in consequence introduced into the subject of the report further. The sandstone of Essen placed at my disposal was not sufficient for the frost test, and in addition was not fresh.

The Frost Test.

Prof. A. Hanisch kindly consented to undertake carrying out this test for the sandstones 2—9, being chairman of the Committee for solving problem 7. The process adopted was that used at the industrial school at Vienna, and it certainly deserves the title which it enjoys of «Severe Frost test». Of particular importance in carrying it out is that the test-pieces (in this case cubes of 6 cm) after being thoroughly saturated with water should be frozen at 17° C. and thawed again 25 times, the conditions being approximated to those occurring in nature, but much more exacting. The results of the frost tests are juxtaposed in table 1, which at the same time provides information regarding the amount of water absorbed by the sandstones. The table shows that all the sandstones submitted to the test, namely samples 2—9, withstood it, sometimes excellently and sometimes well, that is to say, as the test was an unusually severe one the result was that some of the samples showed a high degree and some a satisfactory degree of resistance to frost.

The Hils sandstone is the least satisfactory of the series, though even here the injury caused by the freezing was slight, and only observable after half the tests had been carried through. That the observed alterations in the cubes tested by the freezing process were quite unimportant, is shewn by the comparison with other stones recognised as being frost proof, for example, high class granites, as also by the amount of injury which stones not capable of resisting frost, namely calcarious sandstones, limestones and sandstones, sustained. Similar comparisons can be made by ascertaining the loss of weight. This shows that the greatest loss of weight, column 7 of table 1, that of the Stadtoldendorf sandstones and Hils sandstones, is only a little greater than in the case of many granites, as for instance, the frost-resisting Baveno-granite (0·209 %). Further data regarding the injuries effected by frost are to be found in the work by Hanisch on «Frost Tests with Building Stones of the Austro-Hungarian Monarchy, G. Graeser, Vienna 1895». Further, the loss of weight, in so far as due to the effect of frost, is much smaller than the figures given, because the latter, in addition to the loss in soluble substances, include the materials carried off by the frost test. As regards the Stadtoldendorf sandstone, it may be observed that if a larger number of test-cubes had been tried the loss of weight would have probably been smaller. In the case in question the mean (= 0·349) is that of two very different weight losses, namely 0·645 and 0·054 (the behaviour of the Mehle sandstone is similar). In such cases I am disposed to give more credence to the smaller number, and to attribute the larger figures to hidden imperfections in the material, such as cracks due to rough handling in transport or careless working up, which have had the effect of increasing the loss of weight due to the frost test. Finally the resistance to frost of our sandstones was tested by the softening co-efficient introduced by Tetmajer, or rather by the permanence co-efficient of the same experimenter. Compare here Prof. L. Tetmajer communications of the Institute for Testing Materials to the Swiss Polytechnic of Zurich, vol. 1, Methods and Results, etc., Zurich, 1900. If β_t in t/cm² (also in kg/cm²) represents the specific resistance of material dry., and β_n^* in t/cm² (or also in kg/cm²), the specific resistance of the material wet after 25 freezings, according to Tetmajer the loss of cohesion through the

Table 1.

No.		Description of stone	Description of the test-pieces	Weight of 10 cm³ in kg		Strength dry in kg/cm²		
					Mean		Middle	layer
1	2	Hilssandstone	2a	1·99	1·98	613	482	at commencement
			2b	1·97		495		after frost test
			2c	1·99		468		
2	3	Stadtoldendorf Sandstone	3a	2·15	2·14	653	618	at commencement
			3b	2·16		622		after frost test
			3c	2·11		614		
3	4	Nörten Sandstone	4a	1·99	1·97	402	342	at commencement
			4b	1·95		365		after frost test
			4c	1·96		321		
4	5	Hardegsen Sandstone	5a	2·04	2·04	560	538	at commencement
			5b	2·04		509		after frost test
			5c	2·05		567		
5	6	Osterwalde Sandstone	6a	2·15	2·16	898	1050	at commencement
			6b	2·16		1024		after frost test
			6c	2·16		1075		
6	7	Mehle Sandstone	7a	2·16	2·16	1015	1035	at commencement
			7b	2·17		1105		after frost test
			7c	2·17		965		
7	8	Wrexen Sandstone	8a	1·98	2·01	507	454	at commencement
			8b	2·07		523		after frost test
			8c	1·98		384		
8	9	Obernkirchen Sandstone	9a	2·06	2·10	500	537	at commencement
			9b	2·05		573		after frost test
			9c	2·19		899		

No single test piece was so injured by the frost

Water absorption in weight %		Water absorption in space %		Loss of weight by frost test in % of the original dry weight		Behaviour and appearance after freezing 25 times
	Mean		Mean		Mean	
—	—	—	—	—	—	—
8·60	8·44	16·94	16·70	0·266	0·345	After the 16th freezing a small crack was formed along the edge of a layer but does not extend further. At the end 2 corners were somewhat rounded of after the 25th freezing both corners were a little injured. After the 14th a little crack was noticeable in one corner.
8·27		16·46		0·345		
—	—	—	—	—	—	—
5·04	5·51	12·89	12·76	0·645	0·349	Unaltered by frost
5·98		12·62		0·054		Ditto
—	—	—	—	—	—	—
8·91	9·02	17·34	17·63	0·278	0·244	All edges somewhat rounded off by the frost
9·13		17·89		0·210		Ditto
—	—	—	—	—	—	—
6·13	6·13	12·51	12·51	0·099	0·099	Unaltered by frost
6·10		12·51		0·100		Ditto
—	—	—	—	—	—	—
4·08	4·09	8·81	8·84	0·041	0·036	Unaltered by frost
4·10		8·86		0·031		Ditto
—	—	—	—	—	—	—
3·32	3·24	7·20	7·03	0·020	0·071	Unaltered by frost
3·16		6·86		0·122		Ditto
—	—	—	—	—	—	—
7·15	7·36	14·80	14·90	0·076	0·087	All edges somewhat rounded off by the frost
7·57		14·99		0·098		Ditto
—	—	—	—	—	—	—
5·20	5·17	11·39	11·27	0·060	0·055	Unaltered by frost
5·14		11·15		0·050		Ditto

as to render the pressure test was impossible.

combined water and frost effect is represented by the co-efficient $\chi^* = \frac{\beta_n^*}{\beta_t}$. This gives an explanation of the presumable behaviour of the material in frost. For example in the case of a Hils sandstone, according to column 5 of table 1, $\beta_t = 613$, β_n though unknown is in any case somewhat $< \beta_t^*$, that is to say less than the strength dry after the freezing test, in this case therefore < 482 kg per cm^2. It follows therefore that $\chi^* < \frac{482}{613}$ or $\chi^* < 0{\cdot}79$, while in the case of very frost-proof sandstones $\chi^* = 0{\cdot}5$ and even lower values. The result $\chi^* < 0{\cdot}79$ is in one direction indefinite — as a very slight diminution would mean a great falling off in consistency in the frost-test — but still the true value of χ^* will not differ much from 0·79 and will never be as low as 0·5.

According to the above it may be considered as proved that the various samples of sandstone 2—9 belong to the second vertical column of my test scheme (compare the resistance to weather of «building stones» page 179). At any rate the title does not require alteration, or may at most be completed by the addition of the word «small», for the purpose of qualifying the extreme view previously taken. Of the three samples of stone which were not subjected to the frost test but were subjected to the other tests, namely 10 quartzite, 11 Odenwald syenite and 12 Carrara marble, the assumption was made that they belonged to the same vertical column as Nos. 2—9, i. e. are fully frost-proof. Though in the case of the Essen sandstone must this not be said, on the ground of the weathering observed in the case of the Essen Minster. Possibly this material should be introduced between the two vertical columns of the test table. Unfortunately it is not possible to determine the extent to which the causes, whether natural agents or the effects of frost, really contribute to produce this effect. According to the scheme and the explanations which accompany it, the sandstones 2—9 were materials of this kind, and should be further treated according to the results of the tests. Accordingly

2. I undertook the artificial or abbreviated special weather test corresponding to the action of smoky air, in accordance with the method already proposed by me, with the 11 different samples of stone Nos. 2—12, and in addition with No. 1. The arrangement of the bottle battery is shown in the illustration. The samples of

the stones Nos. 1—12 shaped 70 × 30 × 8·5 mm were suspended on glass rods by means of platinum wires in the bottles, which were connected together by india-rubber tubes. The experiment was conducted in the same way and by the same method as that I previously adopted in order to test the effects of CO_2-O-H_2O, excepting that in addition SO_2 gas was also present, and led through the bottle battery, and that the introductory time of experimenting was much shorter.

The CO_2 and O were obtained compressed in the usual iron cylinders; the SO_2 came from a bottle containing 1 liter of liquid SO_2. The introductory period of experiment occupied 209 hours; the amount of CO_2 used was 1350 liters of the gas; the amount of O was 125 liters of the gas; and SO_2 205 liters of the gas.

The experiment commenced after the plates had been dried and weighed on Aug. 19 1903. The test plates, out of consideration for the Carrara marble exposed with them, could only be immersed a very short time, i. e. 2 to 10 minutes, in the testing fluid (H_2O saturated with CO_2O and SO_2). In spite of this the Carrara plate displayed very marked alteration. The portions that came off, either in the fluid itself or in removing from the bottles and in washing, were dried and weighed. The losses in the case of No. 4 and No. 12 were particularly large. The Norten sample was also very seriously attacked and towards the end of the test displayed buckling, cracking and loss of sand, the loss of sand being increased on drying and weighing. The testing solutions had, with the exceptions of Nos. 10, 11 and 12, turned bright yellow or greenish. In the case of Nos. 3, 7 and 9 there was no coloration, or only a very faint one. The drying was carried out, as in the case of my tests with slates, by means of a wire drying frame. The cooling down was, however, not performed on a large bell glass, but in separate small driers. This method proved to be essential owing to the highly hygroscopic character of the sandstones which, when placed in a common bell glass, damped one another. The drying took place in all cases at a temperature of 125° C. until constancy of weight was attained. This temperature had proved itself suitable to prevent the period of drying being too long. For close grained stones like slate, 105° C. is sufficient. The residues in the testing vessels were very insignificant with the exception of Nos. 4 and 12, and were too slight to be weighed, although observable. Nothing of the kind was observable in the case of Nos. 10 and 11, particularly the former.

The causes of the falling off of sand in the Nörten sandstones are mainly due to calcareous inclusions, as may be observed to be the case by inspection, which discloses calcareous cementing material in the rock itself. It is the gradual extraction of this material by the CO_2 and SO_2 in solution that causes the isolation of the sand grains and the dropping out of sand. The results of the experiments given in table 2, as obtained with the testing materials, speak very plainly. In the first place the absolute weight losses of the test weights (col. 5) are reckoned in weight percentages of the original weight dry (col. 6), that is to say, this gives «the co-efficient of weathering», as we may call it, of the different kinds of stone. In order to make the results clearer, however, the values given in columns 7 and 8 were obtained by giving the values for the weathering coefficient for the two extreme cases of quartzite and Carrara marble as unity. The ratios thus obtained in columns 7 and 8 give the relative «artificial weathering co-efficients» of the different classes of rocks, or what may be called the artificial weathering co-efficient φ and φ'.

Of interest is the extremely high value of the weathering co-efficient φ in the case of Carrara marble, which was certainly not to be expected when we consider previous experience with this material in statues. An exact experiment with Carrara marble had not been previously carried out any more than in the case of other rocks (with the single exception of slates). The high values obtained with the Norten sandstone were quite unexpected. This stone, when employed for building purposes, has proved itself so durable that the test is shown to be a very exacting one, a remark which is equally true of the frost test in the manner applied. The numbers in table 2 show that all the stones are sooner or later attacked more or less powerfully by air containing smoke gases. The amount of injury is shown by the figures given, which does not however prevent the stone giving very good results in the fresh air where little smoke is present. Naturally the values given in the tables also provide means of estimating relatively how the different stones behave in fresh air with little smoke, though of course the injury effected takes enormously longer to manifest itself.

Of great interest to me was the comparison of the Odenwald syenite, which was attacked to an extent by no means small, with the sandstone. Comparatively speaking, are from

this point of view to be regarded as solid and durable sandstones above all the Obernkirchen, the Osterwald, the Stadtoldendorf and the Mehle sandstones, the high qualities of which have been proved in a number of places in buildings in the Prussian Province of Hanover, Westphalia and the Rhineland; in Bremen, Hamburg, and even abroad, where the Obernkirchen sandstone may be observed in Holland, Denmark and Russia. But the Hils and Wrexen sandstone are kinds which are also largely employed and give good results.

According to the experimental results, table 2, the twelve species of stones may be grouped as follows:

1. Quartzite.
2. Obernkirchen sandstone.
3. Osterwald sandstone.
4. Stadtoldendorf sandstone.
5. Odenwald syenite.
6. Mehle sandstone.
7. Hils sandstone.
8. Wrexen sandstone.
9. Hardegsen sandstone.
10. Sandstone from Essen.
11. Nörten sandstone.
12. Carrara marble.

Table 2.

No.	Kind of stone	Commencing dry weight before test in g	Final dry weight after experiment in g	Loss of weight in g	Loss of weight in % of first dry weight	Artificial weathering co-efficient φ	Artificial weathering co-efficient φ'
1	Sandstone from Essen	47·6424	47·0441	0·5983	1·256	598	$^1/_{11}$
2	Hils sandstone	38·8460	38·7582	0·0878	0·226	108	$^1/_{60}$
3	Stadtoldendorf sandst.	43·1047	43·0420	0·0627	0·145	69	$^1/_{94}$
4	Nörten sandstone . .	38·5232	37·3570	1·1662	3.027	1441	$^1/_4 - ^1/_5$
5	Hardegsen sandstone	35·3920	35·2148	0·1782	0·503	211	$^1/_{27}$
6	Osterwald sandstone .	41·2119	41·1625	0·0494	0·120	57	$^1/_{113}$
7	Mehler sandstone . .	41·0483	40·9640	0·0843	0·205	98	$^1/_{62}$
8	Wrexen sandstone . .	36·5145	36·3741	0·1404	0·385	183	$^1/_{35}$
9	Obernkirche sandstone	36·4510	36·4218	0·0292	0·080	38	$^1/_{170}$
10	Quartzite	51·2650	51·2639	0·0011	0·002	1	$^1/_{6304}$
11	Odenwald Syenite . .	54·4348	54·3274	0·1074	0·197	94	$^1/_{69}$
12	Carrara marble	49 1662	42·4756	6·6906	13·608	6480	1

In general the above results apply to the resistance of the stone to all weathering influences, including frost (but neglecting other temperature effects), provided that the atmosphere is assumed to contain a certain amount of smoke. Exceptions, however, are provided by the Carrara marble and the «sandstone from Essen». The latter, owing to the effect that frost has upon it, ranks lower than the Carrara marble, though the way in which it is attacked by the smoky atmosphere of Essen must have greatly facilitated the action of frost upon it. The Carrara marble is best referred to a special group (IIIa). As regards the subdivision of the materials Nos. 2—11 in the groups according to quality 3 and 2, it is clear that the Obernkirchen, Osterwald and Stadtoldendorf stone belong to group 3, while the Nörten stone is to be reckoned as belonging to group 2. The rest is dependent upon the weather co-efficient, whether one takes it at the lowest value as for group 3 or for highest value according to group 2. That this is to a certain extent arbitrary is obvious, as was expressly stated. This difficulty however can be got rid of by an international agreement based on practical experience. To express the situation. more accurately: The artificial weather co-efficient for group III will have to be taken as that of a widely distributed, easily-procurable building stone, the natural weathering co-efficient of which practical experts agree to be small. Even here opinions may differ, though agreement seems possible as soon as results are procurable by which artificial weathering tests can be compared with the natural results obtained from buildings in which the stones have been for a long time in use. In my opinion granites of medium quality should be first considered. In this case use should be made of what has immediately preceded and thus the artificial weathering co-efficient of the Odenwald syenite, which lies closest to the categories of stones referred to, must be taken as lowest figure for group III. Correspondingly the Mehle sandstone is the first member of group II, and the other stones being properly classified are shown in table III.

Total results of tests.

Table 3.

1. Frost test	Produces very great to moderate alterations	Produces none or small alterations		
	Sandstone from Essen	Quartzite, Odenwald Syenite, Carrara Marble and all the tested Sandstones with the exception of sandstone from Essen		
2. The obligatory re-agent test	Usually omitted (here carried out)	Effects marked alteration in		Effects only small alteration in
		1 Mehle sandstone	Carrara Marble	1 Quartzite
		2 Hils sandstone		2 Obernkirchen sandstone
		3 Wrexen sandstone		3 Osterwald sandstone
		4 Hardegsen sandstone		4 Stadtoldendorf sandstone
		5 Nörten sandstone		5 Odenwald Syenite
	General valuation by 1 only	Special valuation by 2 as above		
Quality group	I or I/II	II	IIIa	III

As final result of the examination carried out under I and II is shown:

III. The final valuation of the stones 1—12 as illustrated by table 3

corresponding with the system of testing proposed by me (L. C. p. 179).

IV. Proposals for improving the artificial or shortened weather-resistance tests.

The cause and the results of the tests for resistance to weather described above, tests not reaching very far and circumscribed in effect, show in what direction future repetitions of such experiments in an improved form may be carried out; and I think the testing for resistance to weather may be carried a small step forward.

1. The frost test. The frost test should be carried out in the manner practised hitherto, in accordance with the resolutions of the international conference; the following alterations are, however, suggested:

a) First, as many as possible, at least 10, test-cubes of the prescribed size along the edge (cf. the method of measurement proposed by the mechanical technical Testing Institute at Groß-Lichterfelde near Berlin), should all be sawn out of the same block.

As many as possible should be taken so as to remove discrepancies and differences from the mean value, and as far as possible render inequalities in the manner in which the cubes are prepared unimportant. Further, it should be recommended to test not the strength of the material when dry, but when wet, after finishing the frost test, for the purpose of ascertaining the Tetmajer «constancy co-efficient».

b) The loss of weight on the frost test should be determined as accurately as possible, that is to say, the loss of weight caused by the water during the experiments should be eliminated. Accordingly, the atmospheric air is to be excluded as much as

possible, so as to eliminate the effects of CO_2, O and H_2O, or, what appears more troublesome, to ascertain the loss of weight owing to the reagents during the frost test and to subtract it from the total loss of weight during the frost test.

2. As regards the reagents to be employed, the following improvements are to be recommended:

a) The form of the test plates for the test of the reagents can be retained, 70×30 mm. The thickness should be taken at 8.5 mm since Carrara marble is tested at the same time, and also other samples that are seriously attacked.

b) Instead of exposing only two sample stones as comparisons with the others, namely Carrara marble and quartzite to obtain the extreme values in either direction, it is to be recommended that three samples should be employed. The choice of the third sample, which should be the lowest value for group 3 or the highest value for group 2, must be determined by future considerations, such as are given under II.

c) The drying of the plates must depend upon the composition (hygroscopic qualities) of the material to be tested taken at a suitable drying temperature (before and after the tests with the reagents) to be determined by preliminary experiment.

d) The manipulation of the cooling down of the test plates, according to the separate layers, is to be undertaken separately, that is, the treatment is to be apportioned according to the structure of the material.

e) In any case the three gases CO_2, O and SO_2 are to be allowed to act interchangeably upon the test-plates during the day on which the test is made, the order being CO_2, O; SO_2, O; CO_2, O; and so on. By following this principle, in which, as our experiment showed, no difficulty was encountered, the most practical results were obtained.

f) As regards the effect of the reagents CO_2, O and SO_2 it would appear advisable to employ volumes corresponding to the chemical nature of the objects to be tested, and to obtain an approximate result by the means given in the introduction L. C. pp. 50 to 56. In the case in question this corresponds fairly with the ratio of the volumes of the gases passing through the apparatus,

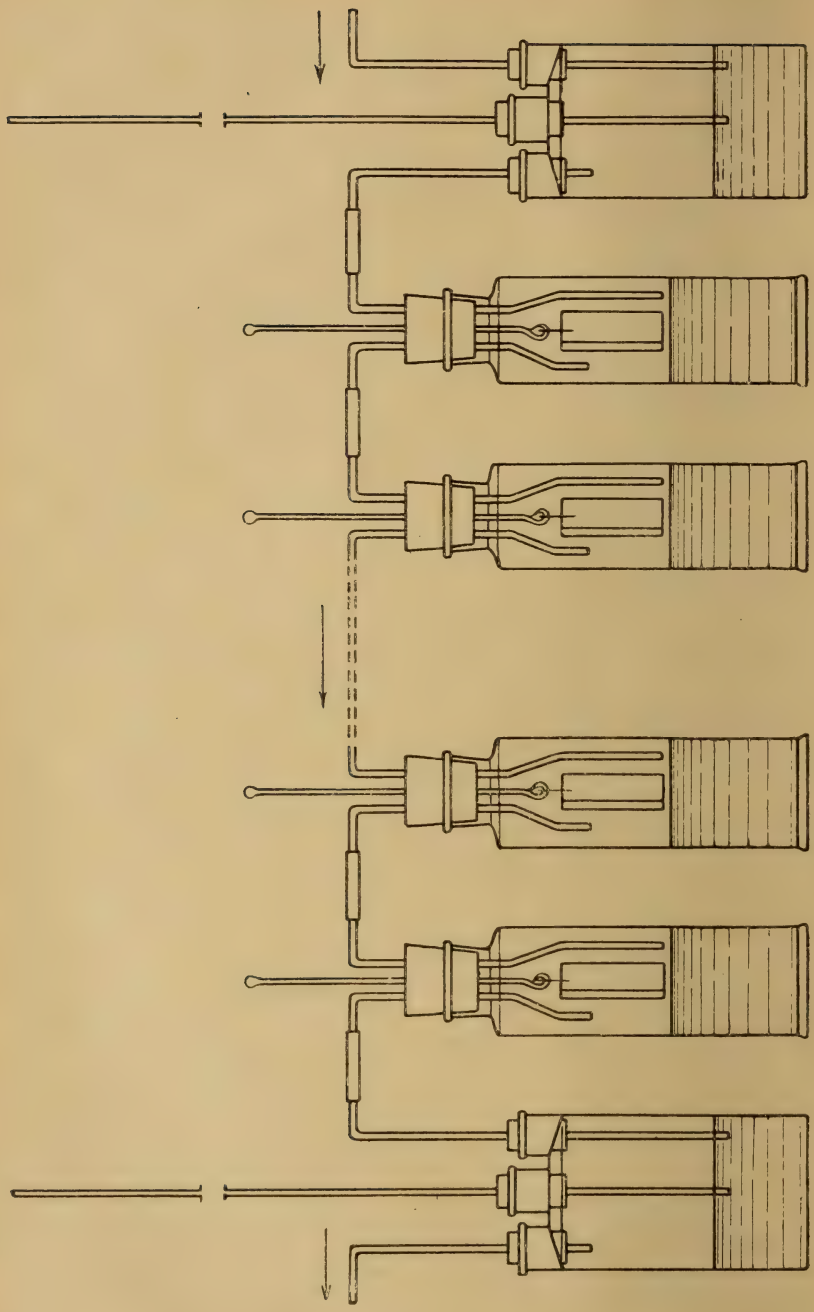
though great excess of CO_2 was used, which however was not prejudicial but only acted as useless ballast. An eighth to a tenth of the actual amount of CO_2 employed would have been sufficient. As the space occupied by the three gases is variable, for strict accuracy and to obtain truly comparative results the same volume of SO_2 gas ought to be employed in each experiment. The International Commission should settle the amount. This is one of the very next questions which should be considered by the International Committee for testing materials for technical purposes. I merely desire to have given the impulse towards the solution of this problem.

The artificial weathering experiments show that the amount of SO_2 as gas which was employed by me was considerably too high, assuming that Carrara marble was among the bodies tested, as it always ought to be. In the test in question the amount of SO_2 gas present was fully sufficient to produce very considerable loss of weight in Carrara marble, and even in a number of other samples of rocks. (Compare columns 5 and 6 of table 2.) In view of the marked destruction of the Carrara marble and of the Nörten sandstone, smaller action by the gas would have been more convenient. On the other hand it is to be remembered that any serious diminution of the amount of SO_2 used, and of the other gases, would still further diminish the amount of weathering exhibited by the quartzite, an error which should be avoided. The amount of SO_2 employed in our experiment would appear to be just right, and I propose accordingly in future to employ approximately 200 liters of SO_2 as gas. In this determination the period of experiment should be diminished as much as possible, and 9 days only should be occupied instead of the previously proposed 36 days. Naturally the proper proportion for the amounts of the three gases must be preserved as closely as possible. It would also be well to adopt a uniform rate for passing the gas through the liquid while keeping the amount used as constant as possible.

In conclusion I cannot refrain from begging the members of the Association, and particularly the managers of the testing institutse, to carry on the experiments commenced by me, and especially

on materials the behaviour of which is known from long continued observation. Only by prolonged and untiring experimental work, hand in hand with practice and experience, can the study of resistance to weather be really carried further.

Special thanks are due to the Committee of the International Association for taking over the greater part of the expense which I have incurred in carrying out my experiments.



Annex IIa*).

Results from Trials on Swiss Roofing-Slate, together with some Importations.

a) The Formation and Texture of Clay-Slate.

By Prof. **Dr. Alb. Heim.**

Translated by Ch. Capito, London.

Of all the rocks of the clay-slate series, the most important for technical purposes is that which is used for roofing, and which has therefore been named «Roofing-Slate». In order to understand the modifications, transitions and occurrences of these rocks, it is necessary to consider the whole series of rocks of which they only form a member.

Clay-Slates are continually formed on the earth. In Nature's large laboratory they require such long ages to be made into finished roofing-slate that we only find them in rock-masses belonging to far remote periods of the earth's history. In reviewing all that has taken place in the whole series of rocks which develop into roofing-slate, we meet with all the stages which have been passed through during the natural manufacture of this material.

The basis is clay. Clay is a soft and plastic sedimentary rock which absorbs and combines with 70 per cent of water and

*) At the desire expressed in several quarters of the International Association, these papers already published in the Communications of the Swiss Federal Institute for Testing Materials (Eidgenössische Materialprüfungsanstalt) at Zurich in 1900 have been reprinted here.

consists mostly of deposits from rivers. When dried at 100°, its specific density is from 2·4 to 2·5. The clays are natural, fine, pulverised washings of decayed and weathered rocks. As a rule, the chief substance is kaolin or kaolinic mineral, that is to say a hydrated silicate of alumina. They also contain particles of minerals which are not weathered or not quite weathered, and which have been carried off by the water. The minerals are principally quartz and mica, often felspar, and also small organic remnants. If lime is precipitated when dissolving clay in water, then the clay is calcareous. There are also dolomitic, limonitic, etc. modifications. The smaller the particles are, the tougher, the more plastic, sticky and «fatter» is the clay, and it is the «leaner» and sandier, the coarser the particles are.

As clay is a washing of weathered rocks, its chemical composition varies considerably. The only thing which is constant about its composition is, that there is always a basis of hydrated silicate of alumina. Silica can also occur under different forms. Ordinary clay contains from 45 to 75 per cent SiO_2, and from 12 to 25 per cent Al_2O_3. The other constituents occur very irregularly.

The young clays when examined under the microscope prove to be throughout of an clastic-pelitic nature, i. e. they are formed of pulverised rock, exclusively of mineral granules which adhere to each other on account of their smallness, but they are not crystalline, nor are they held together by any binding agent.

Progressing from the older argillaceous rocks, the following members will be found in all their conceivable transitions and in all possible combinations with each other:

1. Clays. 2. Shale. 3. Clay Slate. 4. Micaceous Clay-Slate (Phyllite). 5. Mica-Schist.

The transformation consists in the granules perpetually forming denser layers and the texture of the rock becoming more and more laminated or foliated, as the smallest grains gradually re-arrange themselves and become crystalline. The layer of granules becomes slowly a microcrystalline layer, and finally a visible crystalline layer. The small crystals felting together produce greater strength, and finally the schist becomes «bell-hard». Its specific density increases with its compactness from 2·64 to 2.95.

This transformation is principally due to crystallic forces which exist in the substance The necessary factors are time and pressure. We generally find the progress of transformation the greater, the older the rocks are, and the higher the pressure to which they have been subjected. With regard to the pressure, we must distinguish between two kinds, viz: simple pressure due to the dead-weight of younger superincumbent rocks, and the pressure due to the process of Mountain-building. We shall see further on, that the texture of the rock will vary according to the kind of pressure. The process of induration and crystalline transformation of the series «clays . . . . . mica schist» may also be furthered by contact with eruptive rocks, but I do not intend to further discuss the contact-metamorphism, as it hardly ever acts except in narrow limited spaces.

I will first of all endeavour to characterise the principal members of the series according to their constitution.

Shales are soft argillaceous laminated rocks with imperfect lamination; they often fracture in a direction oblique to that of the lamination, the ring is dull, the layer, when examined under the microscope, appears to be still prevalenty indistinctly clastic. As with the clays, there exist varieties which are more or less calcareous (marl-slate), more or less silicious, ferriferous, etc. Also many cement stones.

The Clay-Slates are harder (hardness from 2 to 4), perfectly schistose, and the sheets have a metallic ring when struck. With the naked eye, the substance seems homogeneous and under the microscope it often shows a felting together of crystalline needles and scales, which are often bound together by a colourless silicate of alumina not yet well known. The miscroscope reveals small crystallic elements such as quartz, rutile in needles up to 0·02 mm long, and amounting to about 2 to 3 per cent of the rock, also mica in large quantities of small scales; sericite (a dense, fine-scaly, and mostly green transparent and greasy, soft modification of alkali-mica), chlorite, ottrelite, andalusite, staurolite, hornblende, tourmaline, small grains of pyrite (iron sulphide), haematite (red iron-ore), magnetite (magnetic iron-ore), black scintillating particles of anthraconite (carbonaceous substance) and microscopic scales of calcite. Many constituents are not yet determined with certainty. The trans-

formations, which have taken place, are partly to be seen or traced in the transitions. The formerly sharply defined quartz-grains are still blended with the surrounding rock particles; sericite is formed from the felspar particles, and haematite and magnetite from the limonite. From the organic detritus floated into the clay are formed the scintillating particles of anthraconite, which corresponds to a transformation of organic matter into anthracite or graphite, and, owing to its dark colour and exceedingly fine divisions, the rock is often coloured black. Only those clay-slates which are free from anthraconite are otherwise coloured. Clay-slates rich in sericite are green, and those containing much haematite or silicates of iron are reddish, etc. Close, white clay-slate also occurs. Some of the above named minerals of clay-slate, for example the ores, can also segregate in larger visible grains.

The different modifications of clay-slate contain from 45 to 70 per cent (average 59 per cent) of SiO_2, from 12 to 25 per cent (average 20 per cent) of Al_2O_3, on an average about 7 per cent $Fe_2O_3 + FeO$ and 3-5 per cent K_2O.

With regard to chemical combinations, there are as many varieties of clay-slates as there are of clay and shales. There are calcareous modifications, such as calcareous schist and marl-slate; there are also some which are rich in silica; they are whet-Slate and silicious schist.

The clay-slates to be used for roofing must be thin, evenyl fissile, and weather-proof. The chemical and mineralogical analysis does not give much information about the weather-proofness. They should contain as little as possible lime and pyrite. The weather-proofness depends to a high degree on the structure (or rather on the texture), and it may be quite different although the chemical and mineralogical constituents are the same; and it may be equally good, although the chemical compositions are different.

Mica schist (phyllite) is the name given to those modifications of clay-slate in which the crystallisation is already so far advanced that it is beginning to be visible to the naked eye. Microscopical examination also points out many varieties of minerals similar to those of the clay slates. Above all, there is a display of larger and more distinct crystallites of the different forms of mica, sericite and quartz. Besides these, there are minerals which occur very rarely in clay-slates, such as: Garnet, hornblende,

zoisite, staurolite, albite, etc. The phyllites also form a branch (from the calcitic to the true calcareous phyllites).

Many of the schists which are used for roofing, such as the green sericitic schist from the Ardennes and which contains crystals of magnetite, may conveniently be classed with the phyllites. All the transitions which are due to a more completely crystalline formation, in which the original clastic nature has completely disappeared, carry us at last to a group of crystalline, granular, schistose rocks. Besides other varying minerals, they always contain a large amount of mica, and have therefore received the name of mica-schist.

It must however not be considered certain that all mica schists have been formed in this way. We also meet with a series of rocks, corresponding to the increasing percentage of lime in the original clays, viz: the series of mica-schists extending from the calcareous mica-schist to the mica-marbles (Cippolino).

The clays belong as a rule to the present period (alluvial period), the diluvium and the tertiary time; now and then they are older.

The shales are most frequently met with in the formations of the tertiary system and in the complexes of the mesozoic strata (chalk, jura and trias).

The clay-slate is generally found inside the palaeozoic system of strata (carboniferous, Devonian, Silurian and Cambrian).

Phyllite is principally found in the azoic sediments (Cambrian and pre-Cambrian).

Mica-schists follow amongst the phyllites in the group of crystalline schists.

But very remarkable exceptions from the rule show that pressure assists the progressive transformation and compactness more than time.

Some very old silurian clays occur in Russia which are still plastic and purely clastic, exactly like our diluvial and tertiary clays. These clays lie close to the surface and have never been covered and compressed by larger superincumbent sediments; they have therefore remained plastic notwithstanding their great age.

We meet with the opposite in the Alps, especially in those zones which have been subjected to a very considerable compression, due to the process of Mountain-folding. The metamorphism is here much further advanced than we should expect, according

to average occurence and age. It is here that we found the youngest of well-known roofing-slates, viz: the tertiary (oligocene or young eocene) slate-plates of Matt, Engi, Elm in the canton of Glarus, of Attinghausen in Uri and of Taminathal, as well as the jurassic ones of Meiringen. In the inner trough-beds between the inner-alpine centralmassives are found phyllite and mica-schist, which have become perfectly crystalline, and which contain garnet, mica, zoisite, etc. and jurassic petrifactions.

If these examples prove that pressure is more essential than time, then the explanation of why the average transformations progress with time, must be found in the fact that the older rocks have usually been loaded earlier than the younger ones, and that they therefore have been subjected longer to the action of the load-pressure, and consequently also of heating, than the younger ones.

If one compares the individual constituents of this arranged series (progressive and with numerous transitions in all varieties) Clay, shale, clay-slate, micaceous clay-slate (Phyllite), mica-schist, then one finds a tendency to a steady growing denser compactness, and also to the formation of minerals of higher specific density. The water contained in the original silicate of alumina disappears gradually, and the whole rock becomes correspondingly and specifically denser. It is a transformation accompanied with a diminution of volume, which may be the reason why pressure essentially promotes the transformation.

Finally, a quality of clay-slate most essential for us, viz: lamination or foliation, requires a fuller explanation. The phenomenon is much more complicated than it is usually considered to be. We are still far remote from being able even approximately to separate all the details of this question. It is not possible to recognise with certainty the character of the lamination from any single hand-specimen, but we must also examine the material as a rock-formation and see whether we have to distinguish between stratification or lamination, and how the two are related to each other. According to the formation, there are various kinds of lamination. It may sometimes occur in various clay-slates in one direction only, but it may also occur in the same rock in complex directions. For practical purposes, the material must have a tendency to split in parallel planes. We may with safety distinguish between the following types:

I.

Thin-stratification (slabs, straticulate layers) fig. 1.

When the stratification, on account of change or interruption in the deposit, is very thin, so that the thickness of the single layers is only about one cm, or even a fraction of a cm, then it may be considered, for all practical purposes, a »slate«. This kind of fissility may be called »Thin-stratification« (Dünnschichtung, fälschlich Schieferung). Its character is as follows: The layers are parallel to the stratification of the various larger rocks. Petrifactions, ripple-marks, foot-prints, etc. are often found on the surfaces. The petrifactions lie in the planes of the slabs. The thin slab or plate is as a rule not further fissile, its thickness is determined by nature, and, according to its unalterable thickness, it can be used for one or other purpose (roofs, tables, floors etc.). The upper surfaces of the slabs are often regularly different to the underneath surfaces; the slab is on the whole not homogeneous through its entire thickness.

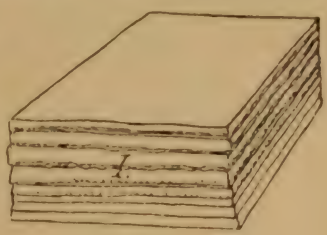


Fig. 1.

Example: «Slates» from Engi and Matt in Sernfthal in the canton of Glarus. In the »Blätterschiefer« the one side is rough, hard, more calcareous and silicious and light grey; the other side is smooth, softer, more argillaceous and graphite black (the »hard side« and the »soft side«). The thickness of the slabs varies from 2 mm to 5 cms. The numerous petrified fishes with still preserved bony matter lie in relief in and on the hard side, the deepest casts are found in the soft underneath-side of the slab above. In this material, we have to do with regular, perhaps seasonable, changes from a more argillaceous to a more calcareous deposit, which has grown into a thin layer. Notwithstanding the considerable amount of lime which they contain, some of the »Plattenschiefer« from Sernfthal make fairly good roofing-slates. They do not flake, they are thin strata, not shale or schist. [See Heim: »Mechanismus der Gebirgsbildung« (mechanism of mountainbuilding), Volume II page 66, etc. and Alex. Wettstein, »Denkschriften der Schweiz. naturforsch. Ges. 1886« — (Memoirs of the Swiss Natural-Philosophical Society)].

II.

True Lamination or Foliation.

True lamination is manifested by a fissile structure which passes through the whole mass of the rock and does not consist only of single select layers. It originates from an arrangement in parallel layers of lamellar or stalky, and as a rule microscopically fine particles of rock. The thickness of the split sheets is not determined by nature, but may be chosen within certain limits. The sheets have no regularly dissimilar sides, but both sides are alike. The lamination may depend on three different causes, and consequently has different characters, viz:

II. A. Original lamination of deposit. (Fig. 2.)

The rock-granules or mud-particles, instead of being in all respects of similar dimensions, may partly or principally be also of a stalky or lamellar (flaky) shape. In this case, they arrange themselves, when deposited from the water and long before the consolidation, more and more horizontally and parallel to the bedding. When flaky granules are predominant, the surfaces of the layers of granules will also be horizontal. For this reason, the adhesion in the horizontal direction will, from the beginning, be less than in the vertical direction; the rock is «laminated» or «foliated».

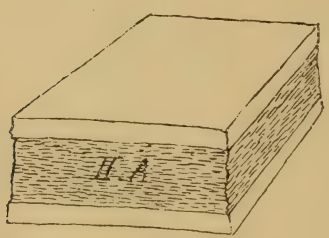


Fig. 2.

The original lamination of deposit occurs most distinctly in psammitic (sandstone) and pelitic (mud) rocks, when the rock from whose weathering and washing it has drawn its building material was very rich in mica. Small mica-flakes then produce principally foliation. They are often visible to the naked eye but also often microscopically small. The greatest formation of original lamination of deposit occurs in the micaceous sandstone, which consists solely of deposited mica-flakes. Good roofing-slates which show only original lamination are very rare indeed.

II. B. „Dead-weight" lamination (consolidation-lamination) (Fig. 3.) is produced by the dead-weight of the younger sediments overlying a clay, whereby all the processes of consolidation will to some extent take place. Instead of contracting uniformly in all directions and forming denser molecular layers and fissile network, the contraction in the vertical direction will be assisted by the dead-weight; in the horizontal direction it will be made more difficult or impossible. The vertical load pushes the material aside which thus becomes continuously more closely interlocked. Whenever a mass is compressed in one single direction, it will become laminated at right angles to this direction, because all chemical, physical, mineral and petrographic irregularities which were primarily present will thereby be squeezed flat into laminae. All original stalky or flaky particles which were irregularly placed will be turned down more or less flat. The diminution of volume through the particles coming together in the horizontal direction will not take place, on account of the squeezing out in the horizontal direction. The dimensions will therefore diminish in the vertical direction and will increase somewhat in the horizontal direction. During this action, the slow transformation from a clastic to a more crypto-crystalline structure becomes more effectual. Thus the mass will necessarily become laminated. This kind of lamination is the most general. It is therefore much more developed and decided in the argillaceous rock-series than, for example, in salt, gypsum and limestone; while the clays which originally contained much water will, during consolidation, diminish their volume at a much higher rate than almost any other rock. Their many lamellar mud-particles assist also in this direction. We thus see, that the older argillaceous rocks almost always become laminated (foliated, schistose). The word „slate" (Schiefer), which only designates a texture, has been used incorrectly as a name for a rock, and often instead of clay-slate only the word slate is used. In short, we might consider this kind of lamination as a consequence of the contraction of argillaceous rocks in the direction of gravity during the process of consolidation.

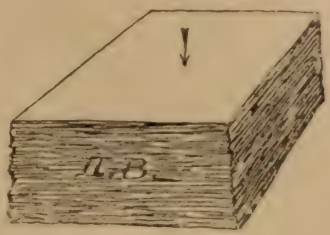


Fig. 3.

The dead-weight or consolidation-lamination is almost or perfectly parallel to the stratification indicated by layers of other rocks, for example beds of sandstone or other modifications of clay-slate. They are therefore on the whole horizontal. The confined petrifactions are imbedded in the direction of the lamination and are as a rule compressed in the direction normal to it. The shales or schists formed in this manner may be solid, but a flaking due to weathering is also possible.

Example: Roofing-slate (clay-slate) from the carboniferous period at Vernayaz-Salvan in Lower Wallis.

II C. „Thrust"-lamination („Pressure-lamination in a restricted sense") „Cleavage", „Squeezed-lamination". (Fig. 4.)

Any mass which is subjected to pressure in one direction while it can extend in another direction, must assume a fissile texture normal to the direction in which the volume is diminishing.

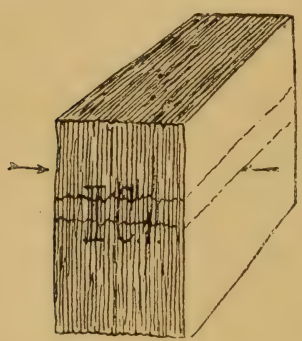


Fig. 4.

The lamination is parallel to the direction of the least resistance, which is always inclined towards, but not necessarily normal to, the direction of the maximum thrust. Thus pastry may become fissile if the dough has been subjected to the process of rolling. Compressed and not very homogeneous manufactured clays (for example pipes, tiles, etc.) be come fissile in burning. Metals which are heterogeneous, or have been rolled at a too low temperature, become laminated. Also quite homogeneous masses form, when compressed, internal slipping surfaces in the direction of flow, and become and remain laminated, unless, by means of another process (welding heat with metals, moisture producing a sticking together with plastic clays, etc.), the cohesion on the internal slipping-surfaces is brought into action again.

Researches made during the last decades have shown that the tangential (horizontal) thrust in the earth-crust, caused by the enormous foldings (i. e. the building of mountain-chains), has compressed and deformed almost all rocks. The prevalent direction of this mountain-pressure is horizontal and it was therefore easiest

for the rocks to flow in an upward direction. The thrust-lamination produced by this pressure is therefore as a rule steep, and the dip is parallel to the mountain-chains. By this process, massive eruptive rocks can be made fissile (gneiss and sericitic schist from granites and porphyrs), it can also transform massive limestones into calcareous schist, and clay-rocks into clay-slate. It promotes the consolidation of clay-slate just as effectively as the pressure from the dead-weight of the superincumbent layers. It may also appear in single hand-specimens quite identical with the dead-weight lamination mentioned in II B. The mountain-pressure, however, often promotes the formation of fissile rocks much more effectively, as in the one direction the pressure is more unlimited and extensive, and in the direction of least resistance the flow can take place to an unbounded amount. What especially distinguishes cleavage from dead-weight lamination is the horizontal direction of the squeezing pressure causing the usual steep cleavage layers.

1. Cleavage or thrust-lamination may occasionally be parallel to the strata and the dead-weight lamination, and thus assist both.

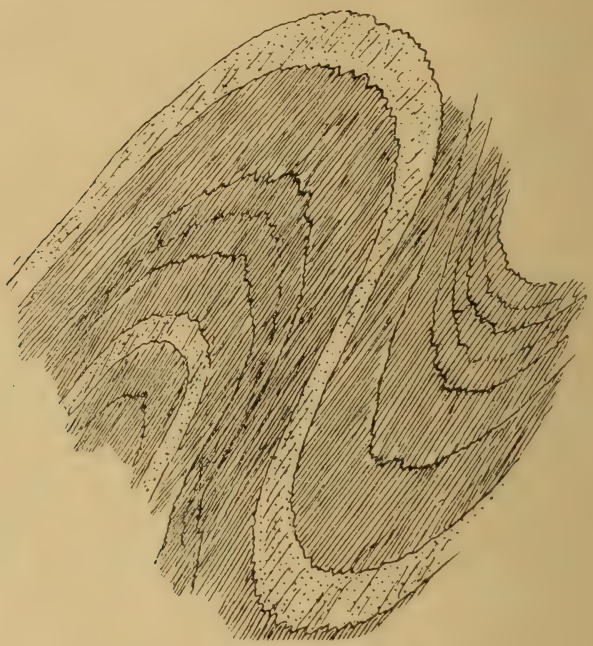
2. The cleavage-planes as a rule intersect the bedding and therefore also the original lamination of deposition as well as the dead-weight lamination: it appears as a transversal lamination, «Slaty-Cleavage,» independent of the line of bedding. As the strata in mountain-chains are folded and the dip therefore occurs in all directions, we find, within a short distance of each other, cleavage which is parallel to the bedding and cleavage which intersects the bedding at any angle up to 90 degrees. Slaty-cleavage is thus the normal form of fissility due to mountain-pressure.

The thrust-lamination when occurring with transversal cleavage-planes possesses the following characteristics:

It has a tendency to split in almost any thickness and the surfaces of the sheets are similar on both sides as in case II, but there are no petrifactions on the sheets. Where such occur, they lie across the cleavage-planes and are much deformed; they are also compressed in a direction normal to the cleavage-planes and are often peculiarly displaced. The lamination intersects the bedding at an oblique or a right angle at the boundary-line of the slate-mass, i. e. at the next stratum. The lines of bedding intersect the cleavage-planes principally near the crests of the folds at a right,

or almost at a right, angle. The strata are here thickened and frilled: they are often cracked and filled with secretions. Where however the lines of bedding intersect the cleavage-planes at an oblique angle, viz. between the crests of the folds, the strata are smooth and thinner, and look as if they had been rolled off (Fig. 5).

Example of clay-slate with pure thrust-lamination: Massive-slate from Elm (canton of Glarus). It is very uniform, splits in thin sheets, is black and fine and produces excellent writing-slates,



Slaty Cleavage in Foldings. (Fig. 5.)

pencils, etc.; it produces however bad roofing-slates as they flake off when weathering.

A very remarkable phenomenon often occurs with thrust-schists, and only with them: On the surfaces of the sheets may be seen more or less distinctly a system of straight streaks in a certain direction which often passes uniformly through the mass of the slate-rock. Many sheets fracture more easily in this direction than across it, and in this case it is important which way the roofing-sheets are cut out of the streaked material. The direction of the streak is in most cases steep i. e. it is almost

parallel to the dip; it thus follows the direction of least resistance of the squeezed matter — in other words the upward direction. When streaks occur together with petrifactions or rubble-stones, coloured patches, etc. the latter will be found to be stretched in the direction of the streaks. Many petrifactions have undergone an extension of 10, 20 or even 30 times their original length, or have been torn to pieces. This is however not the place to enter more closely into all the remarkable phenomena which attend the deformation of rocks due to mountain-building. For our purpose, it is sufficient to recognise that the streaks in the cleavage-surfaces have been caused by the rock-material under pressure having yielded in one direction only, and indicate a direction of flow in the rock. It will be denoted by »Linear-Extension«. This expression is of course not meant to indicate that the rock-particles have been subjected to tension, but that they have stretched in the direction of the least resistance, just as coloured patches in a liquid or in a pulp are stretched into threads by the flow. The linear-extension may go so far in different rocks, also in clay-slates, that the rock divides into four-cornered bars, sticks and pencils, two sides of which are formed by the cleavage-planes and the two others by the linear extension.

We must therefore distinguish between:

1. Cleavage without linear-extension (Fig 4).
2. Cleavage with slight linear-extension
3. Cleavage with distinct linear-extension (Fig. 6).
4. Cleavage with considerable linear-extension.

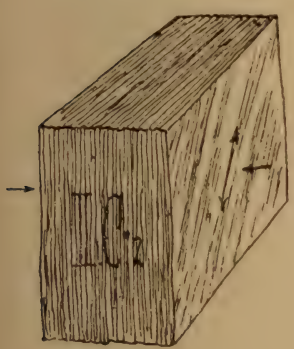


Fig. 6.

Each of these cases may occur when the cleavage is parallel to the bedding as well as when it is transversal.

Finally a great complication may be imagined to which many slates in the Alps bear witness: A primitive cleavage is formed during the first phase of mountain-folding. The progressing mountain-building turns the rock-mass, which has already been transversally laminated, into quite a different position;

thus the new or the continuous thrust once more creates a transversal lamination, which will disturb the first one and cross it, and perhaps finally almost obliterate it. The like transformation may continue.

The cleavage thus formed will at some time in a later phase in turn be traversed by a new one or be broken up. There exist rocks in which are indicated several simultaneous intersecting cleavage-systems in different formations. Good roofing-slate cannot be formed in this manner; it is necessary that a final and enormous compression without a new change of position should take place and in the widest sense transform the rock completely.

Each of the above mentioned fissile structures I, II A, B and C may be so well developed that they will produce good roofing-slates.

III.

Combinations.

The above mentioned different occurrences, which include fissile structures in the widest sense, do not always appear singly. Two or even more may act simultaneously or temporarily after each other on the same rock. Laminated rocks, from which good roofing-slates or writing-slates can be made, will only be found when either:

1. The layers due to the different processes in the same rock are parallel, so as to assist and not disturb each other — or

2. the layers due to the different processes are not mutually parallel, except when one of the processes prevails so as to obliterate the others.

The formation of cleavage is generally the latter process. It very often occurs that the cleavage transforms the rock completely and thoroughly, and obliterates every trace of the older original or dead-load laminations and produces an uniformly good slate. In all cases of combination other than Nos. 1 and 2, it will be impossible to produce good roofing-slates. (Figs. 7 and 8.)

We can easily imagine the variety of textures these combinations may produce in clay-slates. A roofing-slate may often be good at one place and a little further on it may be intersected by another system. Even if the latter is but little developed, the

former will nevertheless be uneven and spoiled, and have a transversal fracture. A closer attention to this subject will certainly clear up many phenomena. We cannot draw up more exact rules than the above named Nos. 1 and 2. It is first of all necessary to test and examine not only the slate itself and where it was long ago formed, but also the quarry and its position in the surrounding hills.

The different kinds of fissility may be tabulated as follows:

I. Thin stratification (parallel layers of thin slabs).

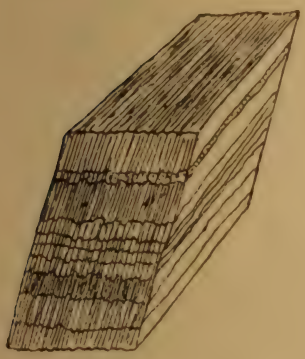


Fig. 7, I and II C.

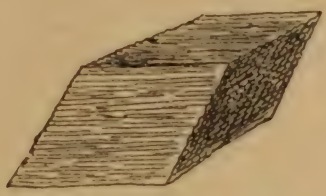


Fig. 8, II B and II C.

II. True Lamination (parallel layers of thin sheets or laminae)
- A) Original Lamination of Deposit.
- B) Dead-Load Lamination (Consolidation Lamination).
- C) Thrust-Lamination (Cleavage, Squeezed Lamination).
  - a) Lamination parallel to bedding.
  - b) Slaty-Cleavage (transversal to bedding).
    1. without linear extension
    2. with « «
       - α) Appearing once.
       - β) Repeated.

III. Combinations.

In conclusion, the following examples of combinations may be cited:

1. Massive slate from Elm in the Canton of Glarus. This slate is formed of a soft clay-slate. The lamination parallel to the

bedding is completely broken up and destroyed. It is now a cleavage slate. The lamination is therefore II B destroyed and superceded by II C b.

2. Pencil-slate from Geisstaffel in Sernfthal. We find here original and dead-load laminations combined with slaty-cleavage without linear-extension. Both are distinctly, and about equally distinctly, formed. The clay-slate rock splits therefore into natural pencils and is used for that purpose. The lamination is therefore II A + II B + II Cb.

3. Pencil-slate from other places in Sernfthal originates from Slaty-cleavage with strong linear-extension, the older laminations parallel to the bedding having been completely destroyed — II B. destroyed by II C. b. 2.

4. Slabs (Plattenschiefer) from Engi in Sernfthal. These are thin-stratifications and are often combined with cleavage parallel to the bedding, and this cleavage shows a very distinct linear-extension, which has stretched the fossil-fishes lying on the surfaces of the slabs. It is thus a combination of thin-stratification (older than the raising of the Alps) with cleavage and linear-extension (due to the raising of the Alps). If we follow the layers of good slabs, we meet at a certain point with a change of character; the material becomes useless because cleavage and thin-stratification intersect each other at an oblique angle which makes the slabs uneven and gives them a transversal fracture. I + II C a 2.

5. Roofing-slate from Salvan possesses a fissile structure which has been caused by the co-operation in parallel directions of the following processes, viz: some original lamination, decided dead-weight-lamination (both older than the Alpine folding), then thrust-lamination which has been formed right through in a vertical direction, in the vertical and squeezed clay-slate, II B + II C a. slightly 2.

6. A great many roofing-slates from Belgium and from Taunus have principally original and consolidation-lamination, cleavage occurs also in many of them, but cleavage together with the destruction of the older parallel laminations only occurs in a few cases.

The favourable cases in which the different systems have supported each other, or in which one of them predominates and the others are obliterated, are much less numerous than the unfavourable and disturbing combinations of clay-slates, which produce

slates useless for technical purposes, and which have a transversal fracture and are ruffled and uneven. Other phenomena also occur, such as splitting, flaws, etc. and which are often not directly connected with the formation of the material. Many good schists have been ruined through the ruffling of the earth-crust by mountain-building; many of them have even been crushed. In the course of time, the schists have been influenced by the greatest variety of circumstances, and few of them have emerged with the uniformity of character which is desired for technical purposes. The earth-crust is full of clay-slate, but the purer, more weather-proof and even kinds are of rarer occurence, and have to be picked out with great trouble.

We may perhaps in time succeed in settling in a clearer manner the relation between the decisive influences and effects during the formation on the constitution and availability of clay-slate.

Annex IIb*).

Results from Trials on Swiss Roofing-Slate, together with some Importations.

Results of the physical-chemical researches

by

Professor L. v. Tetmajer (†).

*) At the desire expressed in several quarters of the International Association, these papers, already published in the Communications of the Swiss Federal Institute for Testing Materials (Eidgenössische Materialprüfungsanstalt) at Zurich in 1900, have been reprinted here.

No. of Sample	Marking of Sample	Material found at	Origin of Sample	Age of Sample	Position of Object w. regard to the points of the compass	Geolog. Age	Colour of Sample	Structural relations
			α) Geological Age, Colour and Structure.					
			Sent in by: Mermoud & Co., Saxon (Valais).					
1	A	Saxon	from Warehouse	—	—	carbon	graphite grey	homogeneous thin sheets
			Sent in by: Jos. Arlettaz, Sembrancher (Valais).					
2	C	Sembrancher	from Warehouse	—	—	carbon	graphite grey	homogeneous thin sheets
			Sent in by: Municipality of Salvan (Valais).					
3	D	Salvan	from Warehouse	—	—	carbon	graphite grey	homogeneous thin sheets
			Sent in by: Department of Public Works of the Canton of Valais, Sion.					
4	B 1	Vernayaz	Sion Barracks	9	South	carbon	slate grey	rich in mica, homog., wavy
5	B 2	»	» »	9	North	»	»	rich in mica, homog., wavy
6	B 3	»	Hotel	17	South	»	»	sericitic, homogeneous, wavy
7	B 4	»	Brigue Ry-station	17	North	»	»	sericitic, homogeneous, wavy
8	B 5	»	Eveché, Sion	56	West	»	»	micaceous, homog., rough
9	B 6	»	» »	56	East	»	»	micaceous, wavy, homog.
0	B 7	Leytron	Eglise de Collège, Sion	8	South	jurassic	»	homog., rough, fairly flat surface
1	B 8	»	Town Hall, Martigny	29	South	»	»	homog., rough, fairly wavy
2	B 9	»	» » »	29	North	»	»	homog., rough, fairly wavy
3	B10	Mex	New College, Sion	4	South	Eoc. (flysch)	graphite grey	homogeneous, flat, smooth
4	B11	»	» » »	4	North	» »	»	homogeneous, flat, smooth
5	B12	Dorenaz	Government Building, Sion	10	West	carbon	»	rich in mica, homog. fairly flat, mod. rough
6	B13	Sembrancher	Town Hall, Sion	15	South	jurassic	slate grey	homog., flat surface, fairly, smooth
			Sent in by: Board of Public Works, Canton of Bern, Bern.					
7	E 1	Fruttigen	Canton Prison, Bern	65	South	Eoc. (flysch)	graphite grey	homog., thin flaky, smooth
8	E 2	»	» » »	65	North	» »	»	—

No. of Sample	Marking of Sample	Material found at	Origin of Sample	Age of Sample	Position of Object with regard to the points of the compass	Geolog. Age	Colour of Sample	Structural relations
Sent in by: Department of Agriculture and Commerce, Canton of Vaud, Lausanne.								
19	F 1	Fumay (Ardennes, France)	Bâtiment à louer, Lausanne	1	East-West	Low. Silur. (Cambrian)	dark-violet	homogeneou even streak
20	F 2	» »	Military Depart., Lausanne	5	»	»	»	homog., eve thin sheets
21	F 3	» »	Cathedral, Lausanne	19	South-North	»	»	homog., even, side rather r
22	F 4	Vernayaz	School of Chem., Lausanne	3	East-West	carbon	light-grey	rich in mic rough, unev
23	F 5	»	School of Physics, Lausanne	14	»	»	blue-grey	sericit., rich in m somew. wavy,
24	F 6	»	Canton Hospital, Lausanne	14	South-North	»	light-grey	rich in mic uneven. hom
25	F 7	Salvan	Hospital Pavillon, Lausann	18	»	»	»	rich in mic homog., eve
Sent in by: Kirchen, Architect, N. E. Ry of Switzerland, Zürich.								
26	G 1	unknown	Ry. Station, Winterthur	15	?	Eocene (?)	graphite grey	even surface, h thin flaky
27	G 2	Alle (Luxemb.)	» » »	15	?	Silur (?)	»	homog., rathe rough, slightly w
28	H 1	Ragaz (Engi?)	Goods-depot Ry. Station, Zürich	20	?	Eoc. (flysch)	grey	homogeneou like No. 3
Sent in by: Albert Bauert, Master-roofer, Zürich.								
29	I 1	Alle (Luxemb.)	from Warehouse	0	—	Silur (?)	dark-grey	pryitic moderat smooth, linear
30	I 2	Rimogne, Ardennes (France)	» »	0	—	Low. Silur	green	sericit. interm. mag., ev., rath. hom., moderat.
31	I 3	Limery-Fumay	» »	0	—	»	violet	slightly linear
32	I 4	Rimogue	» »	0	—	»	light grecnish grey with green. streacks	homog. eve surface, smo
33	I 5	Herbemont (Luxemburg)	» »	0	—	Low. Devon	dark-grey	pyrit., slightly w with linear str
34	I 6	Slate mine Engi*)	from a private house Zürich	15	?	Eoc. (flysch)	graphite	heterogeneou one side even
35	I 7	» »	» » » » »	5	?	»	»	harder than the other s
Sent in by: A. Tobler, District Engineer, N. E. Ry of Switzerland, Zürich.								
36	K 1	Slate mine Engi*)	Coach-house, Mitlödi	17	North	Eoc. (flysch)	slate to graph. grey	rich in mica, hete even surf. like N
37	K 2	» »	Convenience-place, Schwanden	17	»	»	»	as No. 36, on side wavy
38	K 3	» »	Station-building, Nidfurn	17	South	»	»	as No. 36, e surface
39	K 4	» »	Station-building, Luchsingen	1	North	»	graphite grey	as No. 36, o side wavy, kn
40	K 5	» »	Convenience-place, Dießbach	17	South	»	slate grey	as No. 36, e surface
41	K 6	» »	Coach-house, Rüti	17	North	»	»	as No. 3
42	K 7	» »	Station-building, Lintthal	9	»	»	graphite grey	»
43	K 8	» »	Magazine, Ennenda	17	»	»	»	»

*) Quarry: Canton- or Landesplatten-quarry, Engi, canton of Glarus.

Marking of Sample	Material found at	Origin of Sample	Age of Sample	Position of Object with regard to the points of the compass	Geolog. Age	Colour of Sample	Structural relations
Sent in by: Pinösch Brothers, Vulpera.							
L 1	unknown	from Warehouse	0	—	Lias (?)	slate grey	hom., uneven, rough
Sent in from: The Institution for Testing Materials, Zürich.							
M 1	Meyringen	Agricultural-show, 1883	0	—	jurassic (oxford ?)	graphite grey	homog., thin sheets, even, knotty in places
N 1	Stauffacher & Co. Ltd. Engi	Polytechnic, Zürich	20	?	Eoc. (flysch)	graphite grey	heterog., one hard and one soft side, slightly wavy
O 1	slate mine Engi	from Warehouse	0	—	»	dark graphite grey	as No. 16, even surface, mod. rough
O 2	slate quarry Co., Engi	» »	0	—	»	slate grey	rich in mica, flat on one side, otherwise as the last one
O 3	slate mine Elm	» »	0	—	»	»	homog., flat surface, moderately smooth
Sent in by: Canton-Engineer Gremaud, Friburg.							
P 1	Fruttigen (?)	Préfecture, Friburg	100	North	Eoc. (flysch)	slate grey	homogeneous, thin flaky, even
P 2	Vernayaz (?)	Chancery, Friburg	20	»	carbon	blue-grey	sericitic, rather wavy, homogeneous
P 3	Vernayaz	Gendarmery-Station	15	»	»	grey	sericitic, mod. flat, rich in mica, hom.
P 4	Leytron (?)	Hotel cantonale, Friburg	19	»	jurassic	slate grey	homog., even, thin, flaky
P 5	Vernayaz (?)	Cabanons des grands ponts, Friburg	18	»	carbon	blue-gray	sericitic, somewhat wavy, homog.
P 6	Salvan	Valsanite Gruyère, Friburg	15	»	»	slate grey	homogeneous, rich in mica, even
P 7	Sembrancher (?)	Pensionnat de Fribourg	?	»	»	«	homogeneous, one side even
P 8	Mex (?)	Ry-Station of J.-S., Flamatt	37	»	Eoc. (flysch	graphite grey	rich in mica, fairly even, homog.
P 9	Salvan? (Vernayaz)	Church, Attalens	37	»	carbon	light grey	rich in mica, rough, fairly even, homog.
P 10	» »	Town Hall, Attalens	41	»	»	grey	
P 11	Salvan	Chapel, Attalens	37	»	»	slate grey	hom., stratic., somewhat micac., even
P 12	Fumay, Ardennes	Ry-Station of J.-S., Flamatt	37	South	Low. Silur. (Cambrian)	dark violet	hom., ev., thin sheets
Sent in by: Alb. Bauert, Master-roofer, Zürich.							
Q 1	Chiavari (Italian)	from Warehouse	—	—	Eoc. (flysch)	graphite grey	homog., even., thin sheets

β. Thickness and surface-condition of the slate.

No. of Sample	Marking of Sample	Average thickness of plates in cms.	State of sample when delivered
	Sent in by: Mermoud & Co., Saxon.		
1	A	0·42	Perfectly intact, clear ring
	Sent in by: Jos. Arlettaz, Sembrancher.		
2	C	0·47	Perfectly intact, clear ring
	Sent in by: Municipality of Salvan		
3	D	0·55	Perfectly intact, clear ring
	Sent in by: Department of Public Works, Sion.		
4	B 1	0·58	Perfectly intact, clear ring
5	B 2	0·47	„ „ „ „
6	B 3	0·82	„ „ „ „
7	B 4	0·78	„ „ „ „
8	B 5	0·48	„ „ „ „
9	B 6	0·52	„ „ „ „
10	B 7	0·75	„ „ „ „
11	B 8	0·82	„ „ „ „
12	B 9	0·70	„ „ „ „
13	B 10	0·60	„ „ „ „
14	B 11	0·66	„ „ „ „
15	B 12	0·45	„ „ „ „
16	B 13	0·74	Split along the eaves-edge, here and there dull ring.
	Sent in by: Board of Publics Works, Berne.		
17	E 1	ab. 0·95	much weatherad, soft, flaked
18	E 2	ab. 0·95	„ „ „ „
	Sent in by: Department of Agriculture and Commerce, Lausanne.		
19	F 1	0·32	Perfectly intact, clear ring
20	F 2	0·30	„ „ „ „
21	F 3	0·57	„ „ „ „
22	F 4	ab. 1·2 (?)	„ „ „ „
23	F 5	ab. 1·1 (?)	„ „ „ „
24	F 6	ab. 0·7 (?)	„ „ „ „
25	F 7	0·56	„ „ „ „
	Sent in by: Kirchen, Architect, Zürich.		
26	G 1	0·40	Soft on surface, weathered, without flaking, moderately dull ring
27	G 2	0·45	Surfaces lighty weathered, not tender, clear ring
28	H 1	0·82	Perfectly intact, clear ring*)
	Sent in by: Albert Bauert, Zürich.		
29	I 1	0·40	Perfectly intact, clear ring
30	I 2	0·34	„ „ „ „
31	I 3	0·28	„ „ „ „
32	I 4	0·36	„ „ „ „
33	I 5	0·30	„ „ „ „
34	I 6	0·75	On one side in the act of flaking, moderately clear ring
35	I 7	0·82	Exactly as No. 34
	Sent in by: A. Tobler, District Engineer, Zürich.		
36	K 1	ab. 0·80	Partly split, in the act of flaking
37	K 2	ab. 0·50	Beginning to split, moderately dull ring
38	K 3	ab. 0·45	Split, edges soft at places
39	K 4	ab. 0·85	Perfectly intact, clear ring

*) The sample-plate delivered must have been placed in a sheltered position, for by far the greater part of the slate was decayed and weathered and crumbled into small loose pieces on re-roofing the warehouse.

No. of Sample	Marking of Sample	Average thickness of plates in cms.	State of sample when delivered
40	K 5	ab. 0·70	Much split, in the act of flaking
41	K 6	ab. 0·70	Split, in the act of flaking
42	K 7	ab. 0·70	Split, partly flaked on surface
43	K 8	ab. 0·50	In the act of flaking, moderately dull ring
			Sent in by: Pinösch Brothers, Vulpera.
44	L 1	ab. 1·10	Inclined to flake, intact, clear ring
			Sent in by: The Institution for Testing Materials, Zürich.
45	M 1	ab. 0·40	Perfectly intact, clear ring
46	N 1	0·75	Split in places and flaked, moderately dull ring
47	O 1	0·53	Perfectly intact, clear ring
48	O 2	a. 0·90*)	„ „ „ „
49	O 3	0·35	„ „ „ „
			Sent in by: Canton-Engineer Gremaud, Friburg.
50	P 1	ab. 0·66	Split in places, dull ring
51	P 2	ab. 0·75	Perfectly intact, clear ring
52	P 3	ab. 0·55	„ „ „ „
53	P 4	a. 0·60*)	In the act of flaking, edges tender in places, ring varies
54	P 5	0·6-0·8	Perfectly intact, clear ring
55	P 6	0·42	„ „ „ „
56	P 7	ab. 0·90	„ „ „ „
57	P 8	ab. 0·92	„ „ „ „
58	P 9	ab. 0·85	„ „ „ „
59	P 10	0·6-0·8	„ „ „ „
60	P 11	ab. 0·60	„ „ „ „
61	P 12	ab. 0·35	„ „ „ „
			Sent in by: Alb. Bauert, Master-roofer, Zürich.
62	Q 1	0·22-0·40**)	Perfectly intact, clear ring

*) Thickness of Plates fluctuates between 0·55 and 0·90 cm.
**) The one Plate 0·22, the other 0·40 cm.

No. of Sample	Marking of Sample	Chemical composition of the slate in per cent: SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	$CaCO_3$	$MgCO_3$	$CaSO_4$	FeS_2	H_2O + bit.
		γ. Chemical composition. Average from two Analyses.									
		Sent in by: Mermoud & Co., Saxon (Wallis).									
1	A	58·10	22·72	8·01	—	1·78	1·91	0·39	trace	0·25	5·00
		Sent in by: Jos. Arlettaz, Sembrancher (Wallis).									
2	C	55·24	21·19	6·75	—	1·74	4·49	1·86	0·06	0·58	4·53

No. of Sample	Marking of Sample	Chemical Composition of the slate in per cent									
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	$CaCO_3$	$MgCO_3$	$CaSO_4$	FeS_2	H_2O + bit.
	Sent in by: Municipality of Salvan (Wallis).										
3	D	58·62	24·43	5·69	0·37	0·82	0·25	—	0·07	0·02	4·46
	Sent in by: Department of Public Works of Canton of Valais, Sion.										
4	B 1	60·64	20·48	6·46	0·00	2·13	0·20	0·21	trace	0·10	4·97
5	B 2	-	—	—	—	—	trace	—	trace	0·07	—
6	B 3	—	—	—	—	—	trace	—	trace	0·05	—
7	B 4	—	—	—	—	—	trace	—	trace	0·05	—
8	B 5	—	—	—	—	—	0·16	—	0·00	0·07	—
9	B 6	—	—	—	—	—	0·16	—	0·00	trace	—
10	B 7	56·49	7·15	1·75	0·00	0·99	25·35	3·31	0·32	1·46	2·31
11	B 8	—	—	—	—	—	26·43	—	0·28	1·07	—
12	B 9	—	—	—	—	—	26·43	—	0·28	1·07	—
13	B 10	44·91	16·65	4·56	0·47	2·99	23·39	0·00	0·04	0·43	4·54
14	B 11	—	—	—	—	—	22·86	—	0·10	0·59	—
15	B 12	56·22	24·22	6·79	0·44	1·01	1·28	0·00	0·08	0·28	5·48
16	B 13	49·59	20·77	5·29	0·00	0·81	10·70	2·56	0·07	1·89	5·05
	Sent in by: Board of Public Works of Canton of Bern, Bern.										
17	E 1	37·67	9·91	3·20	0·34	2·23	44·89	0·00	0·12	0·53	3·00
18	E 2	—	—	—	—	—	44·89	0·00	0·17	0·49	—
	Sent in by: Department of Agriculture and Commerce of Canton of Vaud, Lausanne.										
19	F 1	59·28	20·47	8·39	0·00	2·04	0·12	0·30	trace	trace	3·91
20	F 2	--	—	—	—	—	0·40	—	0·05	0·01	—
21	F 3	—	—	—	—	—	0·11	—	0·05	0·03	—
22	F 4	59·07	35·85	4·00	0·47	1·71	trace	0·00	trace	trace	4·30
23	F 5	—	—	—	—	—	trace	0·00	trace	0·02	—
24	F 6	—	—	—	—	—	trace	0·00	0·08	0·28	—
25	F 7	64·28	22·49	4·22	0·00	1·55	0·00	0·00	trace	trace	2·66

No. of Sample	Marking of Sample	Chemical Composition of the slate in per cent									
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	$CaCO_3$	$MgCO_3$	$CaSO_4$	FeS_2	H_2O + bit.
	Sent in by: Kirchen, Architect, N. E. Ry of Switzerland, Zürich.										
26	G 1	31·36	12·00	3·64	0·00	2·51	43·50	0·50	1·19	0·45	4·00
27	G 2	53·01	17·79	7·88	0·00	1·60	5·95	2·86	0·15	0·63	5·23
28	H 1	44·37	15·13	4·21	0·00	0·46	20·29	7·12	0·17	0·73	3·39
	Sent in by: Alb. Bauert, Master-roofer, Zürich.										
29	I 1	54·53	18·11	7·56	0·00	1·91	6·55	2·48	trace	1·21	4·37
30	I 2	58·93	17·82	11·81	0·00	1·93	trace	1·43	0·00	0·06	2·85
31	I 3	60·44	19·26	8·94	0·13	3·25	0·80	0·00	trace	0·21	3·16
32	I 4	58·64	20·37	8·56	1·38	3·74	0·14	0·00	0·00	0·04	3·38
33	I 5	55·67	20·27	5·73	0·00	2·36	5·95	1·00	trace	0·77	4·43
34	I 6	45·08	12·72	4·52	0·00	2·48	25·68	3·46	0·09	0·62	3·04
35	I 7	43·43	14·44	4·90	0·00	2·22	28·57	2·10	trace	0·72	3·10
	Sent in by: A. Tobler, District Engineer, N. E. Ry of Switzerland, Zürich.										
36	K 1	46·56	13·79	3·59	0·00	1·91	23·95	1·16	0·29	0·58	3·29
37	K 2	—	—	—	—	—	26·73	—	0·30	0·57	—
38	K 3	—	—	—	—	—	26·25	—	0·65	0·75	—
39	K 4	—	—	—	—	—	25·84	—	0·30	0·58	—
40	K 5	43·47	13·48	4·49	0·00	2·15	28·29	1·72	0·16	0·73	3·33
41	K 6	—	—	—	—	—	28·13	—	0·14	0·73	
42	K 7	—	—	—	—	—	24·70	—	trace	0·94	—
43	K 8	—	—	—	—	—	25·84	—	trace	0·41	
	Sent in by: Pinösch Brothers, Vulpera.										
44	L 1	24·94	6·65	1·87	0·00	0·93	59·34	2·94	0·24	1·01	1·84
	Sent in by: The Institution for Testing Materials, Zürich.										
45	M 1	49·48	16·78	5·49	0·00	3·17	18·71	0·00	0·43	0·89	5·01
46	N 1	45·47	14·62	4·38	0·00	1·33	25·39	1·32	0·29	0·64	1·43
47	O 1	43·97	14·32	3·16	0·00	2·27	28·49	2·32	0·06	0·66	2·96
48	O 2	44·13	14·80	3·03	0·00	2·01	27·28	3·35	0·04	0·40	2·42
49	O 3	36·46	13·29	2·86	0·07	3·35	39·18	0·00	0·07	0·69	3·64

No. of Sample	Marking of Sample	Chemical Composition of the slate in per cent									
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	$CaCO_3$	$MgCO_3$	$CaSO_4$	FeS_2	H_2O + bit.
		Sent in by: Canton-Engineer Gremaud, Friburg.									
50	P 1	37·23	12·08	3·25	0·00	2·51	39·35	0·27	0·24	0·49	3·16
51	P 2	59·59	25·67	3·14	0·56	1·08	0·00	0·00	0·08	0·03	3·93
52	P 3	57·33	27·44	3·86	0·36	1·18	0·00	0·00	0·09	0·06	4·18
53	P 4	55·95	9·39	1·99	0·00	1.90	22·00	2·96	0·20	1·43	2·97
54	P 5	60·69	23·26	4·43	0·25	1·25	0·00	0·00	0·05	0·05	3·84
55	P 6	61·76	21·18	5·45	0·45	1·66	0·00	0·00	0·05	0·04	3·77
56	P 7	59·02	24·65	4·03	0·28	1·46	0·00	0·00	0·12	0·10	3·98
57	P 8	46·18	14·74	4·45	0·00	1·96	23·91	2·02	0·05	0·40	3·36
58	P 9	58·39	22·66	7·20	0·41	1·50	0·16	0·00	0·07	0·04	3·92
59	P 10	60·28	22·61	4·06	0·26	1·34	1·27	0·00	0·07	0·11	3·85
60	P 11	59·05	25·99	2·67	0·30	1·12	0·11	0·00	0·10	0·03	4·00
61	P 12	60·42	19·18	8·35	0·45	2·27	0·80	0·00	0·06	0·03	3·40
		Sent in by: Alb. Bauert, Master-roofer, Zürich.									
62	Q 1	26·95	10·64	3·36	0·17	1·97	54·43	0·00	0·05	0·11	2·42

No. of Sample	Marking of Sample	Specific density of material		Absorption of Water in weight per cent after		Height of wetting in cms. after 24 hours	Porousness in volume per cent		Behaviour during the Freezing Trial
		absolute	apparent	48 hours	28 days		absolute	apparent	
		δ. Physical qualities of the Slates and their Behaviour, during the Freezing test.							
		Sent in by: Mermoud & Co., Saxony (Wallis).							
1	A	2·83	2·80	0·78	0·83	0·0	1·0	2·4	unaltered
		Sent in by: Jos. Arlettaz, Sembrancher (Wallis).							
2	C	2·82	2·80	0·42	0·60	0·0	0·7	1·6	unaltered
		Sent in by: Municipality of Salvan (Wallis).							
3	D	2·81	2·80	0·17	0·20	0·0	0·4	0·6	unaltered

No. of Sample	Marking of Sample		Specific density of material		Absorption of Water in weight per cent after		Height of wetting in cms. after 24 hours	Porousness in Volume per cent		Behaviour during the Freezing Trial
			absolute	apparent	48 hours	28 days		absolute	apparent	
Sent in by: Department of Public Works of the Canton of Valais, Sion.										
4	B	1	2·79	2·76	0·39	0·42	0·1	1·1	1·2	unaltered
5	B	2	2·78	2·77	0·44	0·52	0·1	0·4	1·4	slight enlargement of the original cleavage-planes
6	B	3	2·81	2·79	0·13	0·17	0·1	0·8	0·5	unaltered
7	B	4	2·79	2·76	0·64	0·72	0·1	1·1	2·0	one test unatered, the other showed cleavage-enlargement after the 10th trial
8	B	5	2·80	2·80	0·12	0·19	0·1	0·0	0·5	unaltered
9	B	6	2·80	2·80	0·09	0·14	0·1	0·0	0·4	»
10	B	7	2·74	2·70	0·54	0·60	0·0	1·4	1·6	»
11	B	8	2·72	2·68	—	—	—	1·4	—	not made
12	B	9	2·72	2·69	0·57	0·60	0·1	1·1	1·6	unaltered
13	B	10	2·75	2·75	0·41	0·43	0·0	0·0	1·2	»
14	B	11	2·78	2·76	0·29	0·32	0·0	0·7	0·9	»
15	B	12	2·85	2·81	1·15	1·21	0·1	1·4	3·4	»
16	B	13	2·84	2·79	1·41	1·74	0·2	1·8	4·9	begins to splinter after the 5th trial, loosening has not taken place
Sent in by: Board of Public Works of the Canton of Bern. Bern.										
17	E	1	—	—	—	—	—	—	—	Freezing test not made as the material was too much weathered
18	E	2	2·73	2·69	0·82	0·89	0·1	1·4	2·4	splitting begins after 20th trial
Sent in by: Department of Agriculture and Commerce of the Canton of Vaud, Lausanne.										
19	F	1	2·86	2·83	0·24	0·26	0·0	1·1	0·7	unaltered
20	F	2	2·85	2·84	0·11	0·13	0·0	0·4	0·4	»
21	F	3	2·83	2·82	0·24	0·32	0·0	0·4	0·9	»
22	F	4	2·77	2·77	0·24	0·26	0·2	0·0	0·7	»
23	F	5	2·80	2·80	0·51	0·56	0·2	0·0	1·6	»
24	F	6	2·81	2·78	0·16	0·17	0·1	1·1	0·5	almost unaltered
25	F	7	2·79	2·78	0·12	0·15	0·1	0·4	0·4	unaltered
Sent in by: Kirchen, Architect, N. E. Ry. of Switzerland, Zurich.										
26	G	1	—	—	—	—	—	—	—	not made due to want of material
27	G	2	2·83	2·78	0·61	0·64	0·1	1·8	1·8	after the 14th trial begins slight flaking off
28	H	1	2·77	2·75	0·52	0·54	0·1	0·7	1·5	unaltered

No. of Sample	Marking of Sample	Specific density of material		Absorption of Water in weight per cent after		Height of wetting in cms. after 24 hours	Porousness in volume per cent		Behaviour during the Freezing Trial
		absolute	apparent	48 hours	28 days		absolute	apparent	
				Sent in by: Alb. Bauert, Master-roofer, Zürich.					
29	I 1	2·82	2·77	0·14	0·22	0·2	1·8	0·6	unaltered
30	I 2	2·88	2·80	0·15	0·28	0·0	2·8	0·8	»
31	I 3	2·85	2·83	0·15	0·17	0·1	0·7	0·5	»
32	I 4	2·85	2·82	0·12	0·18	0·0	1·1	0·5	»
33	I 5	2·79	2·74	0·51	0·63	0·0	1·8	1·7	»
34	I 6	2·78	2·74	0·48	0·49	0·1	1·4	1·3	splitting begins after 8th trial
35	I 7	2·76	2·74	0·56	0·59	0 0	0·7	1·6	unaltered
				Sent in by: A. Tobler, District-Engineer, N. E. Ry of Switzerland, Zürich					
36	K 1	2·77	2·73	0·31	0·34	0·1	1·4	0·9	after 10th trial begins slight flaking off
37	K 2	2·76	2·70	0·72	0·73	0·3	2·2	2·0	after 10th trial begins slight splitting and flaking off
38	K 3	2·75	2·74	0·38	0·40	0·1	0·4	1·1	after 7th trial begins small flaking off
39	K 4	2·77	2·73	0·32	0·35	0·2	1·4	1·0	unaltered
40	K 5	2·78	2·70	0·56	0·59	0·0	2·9	1·6	slightly flaky on one side after 24th trial
41	K 6	2·78	2·73	0·37	0·39	0·0	1·8	1·1	as the last one; one test unaltered
42	K 7	2·76	2·75	0·69	0·71	0 1	0·4	1·0	unaltered
43	K 8	2·78	2·74	0·55	0·67	0 3	1·4	1·8	after 9th trial begins slight flaking off
				Sent in by: Pinösch Brothers, Vulpera.					
44	L 1	2·76	2·69	0·41	0·47	0·1	2·5	1·3	after 5th trial begins flaking off on surface
				Sent in by: The Institution for Testing Materials, Zürich.					
45	M 1	2·77	2·75	0·55	0·58	0·0	0·7	1·6	unaltered
46	N 1	2·77	2·73	0·70	0·76	0·2	1·4	2·1	»
47	O 1	2·77	2·74	0·48	0·57	0·0	1·1	1·6	»
48	O 2	2·77	2·75	0·42	0·46	0·1	0·7	1·3	»
49	O 3	2·77	2·72	0·67	0·69	0·0	1·8	1·9	»
				Sent in by: Canton-Engineer Gremaud, Friburg.					
50	P 1	2·76	2·72	—	—	0·0	1·4	—	not made
51	P 2	2·81	2·79	0·22	0·28	0·0	0·7	0·8	splitting begins after 20th trial
52	P 3	2·83	2·80	0·13	0·16	0·0	1·1	0·5	splitting begins after 20th trial
53	P 4	2·75	2·70	0·81	0·88	0·0	1·8	2·4	splitting begins after 25th trial

No. of Sample	Marking of Sample	Specific density of material		Absorption of Water in weight per cent after		Height of wetting in cm after 24 hours	Porousness in Volume per cent		Behaviour during the Freezing Trial
		absolute	apparent	48 hours	28 days		absolute	apparent	
54	P 5	2·82	2·78	0·16	0·21	0·0	1·4	0·6	unaltered
55	P 6	2·82	2·77	0·18	0·27	0·1	1·8	0·8	»
56	P 7	2·82	2·78	0·33	0·43	0·0	1·4	1·2	splitting begins after the 25th trial
57	P 8	2·77	2·75	0·13	0·30	0·2	0·7	0·8	almost unaltered
58	P 9	2·83	2·80	0·93	1·05	0·0	1·1	2·9	splitting begins after the 25th trial
59	P 10	2·82	2·78	0·47	0·56	0·0	1·4	1·6	unaltered
60	P 11	2·83	2·80	0·22	0·62	0·0	1·1	1·7	»
61	P 12	2·84	2·82	0·32	0·33	0·0	0·7	0·9	»
		Sent in by: Alb. Bauert, Master-roofer, Zürich.							
62	Q 1	2·76	2·73	0·48	0·49	0·0	1·1	1·3	almost unaltered

No. of Sample	Marking of Sample	State of Sample at delivery	Age in years of the material	Preservation hitherto	Behaviour during the Fresenius Weatherproof-Test (sulphurous acid)
		ε. Behaviour of the slate hitherto in the open air, and in the Fresenius Weatherproof-Test.			
		Sent in by: Department of Public Works of the Canton of Valais, Sion.			
1	B 1	Pyritic, perfectly intact, clear ring	9	good	unaltered after 6 weeks
2	B 2	Perfectly intact, clear ring	9	»	» » 6 »
3	B 3	» » » »	17	»	» » 6 »
4	B 4	» » » »	17	»	» » 6 »
5	B 5	» » » »	56	»	» » 6 »
6	B 6	» » » »	56	»	» » 6 »
7	B 7	» » » »	8	»	surface inflation begins after 6 days, splitting begins after 21 days
8	B 8	» » » »	29	»	splitting begins after 22 days
9	B 9	» » » »	29	»	surface inflation begins after 5 days, splitting begins after 11 days
10	B 10	» » » »	4	»	splitting begins after 15 days
11	B 11	» » » »	4	»	» » » 5 »
12	B 12	Slightly pyritic, perfectly intact clear ring	10	»	almost unaltered after 6 weeks, the edge slightly tender
13	B 13	Along the eaves-edge a splitting like in calc-spar, dull ring at places	15	»	splitting begins after 12 days

No. of Sample	Marking of Sample	State of sample at delivery	Age in years of the material	Preservation hitherto of the material	Behaviour during the Fresenius Weatherproof-Test (sulphurous acid)
Sent in by: Board of Public Works of the Canton of Bern, Bern.					
14	E 1	Much weathered, soft, flaked	65	bad	Splittings begins after 5 days
15	E 2	Partly weathered, soft, flaked	65	»	Begins to flake after 3 days splitting after 9 days
Sent in by: Department of Agriculture and Commerce of the Canton of Vaud, Lausanne.					
16	F 1	Perfectly intact, clear ring	1	good	unaltered after 6 weeks
17	F 2	» » » »	5	»	» » 6 »
18	F 3	» » » »	19	»	» » 6 »
19	F 4	» » » »	3	»	» » 6 »
20	F 5	» » » »	14	»	» » 6 »
21	F 6	» » » »	14	»	» » 6 »
22	F 7	» » » »	18	»	» » 6 »
Sent in by: Kirchen, Architect, N. E. Ry. of Switzerland, Zürich.					
23	G 1	Surface soft, weathered without flaking, moderately dull ring	16	doubtful	Splitting begins after 15 days
24	G 2	Surface slightly weathered; not soft, clear ring	16	satisfactory	Unaltered after 6 weeks
25	H 1	Perfectly intact, clear ring	ab. 20	bad	Splitting begins after 3 days
Sent in by: Alb. Bauert, Master-roofer, Zürich.					
26	I 1	Perfectly intact, clear ring	0	—	Almost unaltered after 6 weeks, somewhat tender
27	I 2	» » » »	0	—	Unaltered after 6 weeks
28	I 3	» » » »	0	—	» » 6 »
29	I 4	» » » »	0	—	» » 6 »
30	I 5	Perfectly intact, clear ring, some plates considerably pyritic	0	—	Somewhat tender after 6 weeks, otherwise intact
31	I 6	Almost intact, clear ring	15	?	Inflation and splitting begin after 6 days
32	I 7	» » » »	5	?	Splitting and flaking begin after 6 days
Sent in by: A. Tobler, District-Engineer, N. E. Ry. of Switzerland, Zürich.					
33	K 1	Partly split, in the act of flaking	17	bad	Flaking begins after 2 days
34	K 2	Fairly well preserved, begins splitting with dull ring	17	doubtful	» » » 2 » / Splitting » » 4 »
35	K 3	Split, edges soft at places	17	bad	» » » 3 »
36	K 4	Perfectly intact, clear ring	1	—	» » » 2 »
37	K 5	Greatly split, in the act of flaking	17	bad	» » » 2 »

No. of Sample	Marking of Sample	State of sample at delivery	Age in years	Preservation hitherto of the material	Behaviour during the Fresenius Weatherproof-Test (sulphurous acid)
38	K 6	Split, in the act of flaking	17	bad	flaking begins after 6 days
39	K 7	Split and partly flaked on the surface	9	«	splitting » » 4 »
40	K 8	In the act of flaking	17	»	flaking » » 2 »
					splitting » » 4 »
		Sent in by: Pinösch Brothers, Vulpera.			
41	L 1	Perfectly intact, clear ring, inclined to flake	0	unknown	after 4 days begins the loosening on surface, after 4 weeks begins flaking, surface tender
		Sent in by: The Institution for Testing Materials, Zürich.			
42	M 1	Perfectly intact, clear ring	0	bad	splitting begins after 4 days
43	N 1	Partly split and flaked, partly in the act of flaking	about 20	»	» » » 3 »
44	O 1	Perfectly intact, clear ring	0	» *)	» » » 4 »
45	O 2	» » » »	0	» *)	» » » 4 »
46	O 3	» » » »	0	» *)	» » » 3 »
		Sent in by: Canton-Engineer, Gremaud, Friburg.			
47	P 1	Split at places, dull ring	100	at first good	splitting begins after 10 days
48	P 2	Perfectly intact, clear ring	20	good	unaltered after 6 weeks
49	P 3	» » » »	15	»	» » 6 »
50	P 4	In the act of flaking, edges soft at places, ring varies	19	doubtful	splitting begins after 7 days
51	P 5	Perfectly intact, clear ring	18	good	unaltered after 6 weeks
52	P 6	» » » »	15	»	» » 6 »
53	P 7	» » » »	?	»	» » 6 »
54	P 8	» » » »	37	»	flaking begins after 6 days
55	P 9	» » » »	37	»	splitting » » 8 »
56	P 10	» » » »	41	»	unaltered after 6 weeks
57	P 11	» » » »	37	»	» » 6 »
58	P 12	» » » »	37	»	» » 6 »
		Sent in by: Albert Bauert, Master-roofer, Zürich.			
59	Q 1	Perfectly intact, clear ring	0	unknown	splitting begins after 2 days

*) According to expert's opinion the material is inferior.

No. of Sample	Marking of Sample	State of Sample at delivery	Age in years of the material	Preservation hitherto of the material	Loss in weight per cent	Behaviour during the combined Weatherproof-Test
		ζ Behaviour of the slate hitherto in the open air and during the combined Weatherproof-Test.				
		Sent in by: Department of Public Works of the Canton of Valais, Sion.				
1	B 1	Pyritic, perfectly intact, clear ring	9	good	—	unaltered
2	B 2	Perfectly intact, clear ring	9	»	—	»
3	B 3	» » » »	17	»	—	»
4	B 4	» » » »	17	»	—	»
5	B 5	» » » »	56	»	—	»
6	B 6	» » » »	56	»	—	»
7	B 7	» » » »	8	»	10	splitting begins after the 8th freezing trial; the material still fairly hard after the 25th freezing trial
8	B 8	» » » »	29	»	18	after the 20th freezing trial it begins to be tender, after the 25th freezing trial begins splitting
9	B 9	» » » »	29	»	25	after the 14th freezing trial it begins to be brittle, weak ring; after the 20th freezing trial material totally tender falls to pieces
10	B 10	» » » »	4	»	45	after 12th freezing trial it begins to scale, surface rids of slime becomes tender after 20th freezing trial
11	B 11	» » » »	4	»	—	scaling begins, material tender after 12th freezing trial; material breaks up into sheets after 17th freezing trial
12	B 12	Slightly pyritic, perfectly intact, clear ring	10	»	—	material unaltered after 25th freezing trial
13	B 13	Split like calc-spar along the eaves-edge, dull ring at places	15	»	—	splitting begins after 12th freezing trial material quite tender right through split, pliable, plastic after 17th freezing trial
		Sent in by: Board of Public Works of the Canton of Bern, Bern.				
14	E 1	Considerably weathered, soft, flaked	65	bad	—	available sample failed
15	E 2	Partly weathered, soft, flaked	65	»	—	splitting into sheets begins after 1st freezing trial; complete loosening into sheets after 2nd freezing trials
		Sent in by: Department of Agriculture and Commerce of the Canton of Vaud, Lausanne.				
16	F 1	Perfectly intact, clear ring	1	good	—	unaltered
17	F 2	» » » »	5	»	—	»
18	F 3	» » » »	19	»	—	»
19	F 4	» » » »	3	»	—	»
20	F 5	» » » »	14	»	—	»
21	F 6	» » » »	14	»	—	»
22	F 7	» » » »	18	»	—	»

No. of Sample	Marking of Sample	State of Sample at delivery	Age in years of the material	Preservation hitherto of the material	Loss in weight per cent	Behaviour during the combined Weatherproof-Test
		Sent in by: Kirchen, Architect, N. E. Ry. of Switzerland, Zürich.				
23	G 1	Surface soft, weathered, without flaking, moderately dull ring	16	doubtful	—	edges tender, ring still clear, material firm after 18th freezing trial
24	G 2	Surface slightly weathered, not soft, clear ring	16	satisfactory	2	edges tender, otherwise intact after 13th freezing trial; ring weak, begins to split after 18th freezing trial.
25	H 1	Perfectly intact, clear ring	ab. 20	bad	—	flaking begins after 5th freezing trial; breaks up in sheets, material brittle after 10th freezing trial
		Sent in by: Alb. Bauert, Master-roofer, Zürich.				
26	I 1	Perfectly intact, clear ring	0	—	—	material unaltered after 25th freezing trial
27	I 2	» » » »	0	—	—	material unaltered (only shows a few small cracks on surface) after 25th freezing trial
28	I 3	» » » »	0	—	—	material unaltered after 25th freezing trial
29	I 4	» » » »	0	—	—	material unaltered (ring somewhat weak) after 25th freezing trial
30	I 5	» » » » Some plates considerably pyritic	0	—	—	formation of clefts, material tender, ring weak after 17th freezing trial
31	I 6	Almost intact, moderately clear ring	15	?	—	flaking off on one side after 2nd freezing trial; one side of material tender after 5th freezing trial
32	I 7	Almost intact, moderately clear ring	5	?	—	surface tender after 5th freezing trial; material tender, progressive weathering on surface after 20th freezing trial; material tender lamellar scaling off after 25th freezing trial
		Sent in by: A. Tobler, District-Engineer, N. E. Ry. of Switzerland, Zürich.				
33	K 1	Partly split, in the act of flaking	17	bad	45	loosening into sheets begins after 7th freezing trial; breaks up into sheets, material becoming tender after 17th freezing trial
34	K 2	Fairly well preserved, begins splitting with dull ring	17	doubtful	—	wartlike formation on the surface, splitting begins, surface tender after 7th freezing trial; considerable scaling off, material splitting apart after 10th freezing trial; total decay after 18th freezing trial
35	K 3	Split at the edges, soft in places	17	bad	—	exactly as Sample K 2
36	K 4	Perfectly intact, clear ring	1	»	15	splitting begins, ring weak after 7th freezing trial; material becoming tender after 10th freezing trial; breaks up into sheets after 18th freezing trial
37	K 5	Greatly split, in the act of flaking	17	»	—	wartlike formation, surface splitting after 7th freezing trial; splitting of whole plates, material fairly hard, ring clear after 10th freezing trial; completely split, still hard after 25th freezing trial
38	K 6	Split, in the act of flaking	17	»	—	flaking off on surface after 7th freezing trial; breaking up into sheets after 10th freezing trial; completely broken up, brittle after 25th freezing trial

No. of Sample	Marking of Sample	State of Sample at delivery	Age in years	Preservation hitherto of the material	Loss in weight per cent	Behaviour during the combined Weatherproof-Test
39	K 7	Split and surface partly flaked	9	bad	—	split into large lamellae, weak ring after 7th freezing trial; completely split after 25th freezing trial
40	K 8	In the act of flaking	17	»	—	formation of clefts begins, material becomes tender, weak ring after 7th freezing trial; breaking up into sheets after 10th freezing trial; brittle, right through, tender after 25th freezing trial
		Sent in by: Pinösch Brothers, Vulpera.				
41	L 1	Perfectly intact, clear ring, inclined to flake off	0	unknown	—	surface soft after 7th freezing trial; begins to flake off, weak ring after 8th freezing trial; material becomes steadily more tender after 20th freezing trial
		Sent in by: The Institution for Testing Materials, Zürich.				
42	M 1	Perfectly intact, clear ring	0	bad	—	surface tender after 5th freezing trial; flaking off begins after 8th freezing trial
43	N 1	Partly split and flaked	ab. 20	»	—	surface tender after 5th freezing trial
44	O 1	Perfectly intact, clear ring	0	» *)	—	» » » » » »
45	O 2	» » » »	0	» *)	—	» » » » » »
46	O 3	» » » »	0	» *)	—	» » » » » » quite tender after 6th freezing trial
		Sent in by: Canton-Engineer Gremaud, Friburg.				
47	P 1	Split in places, dull ring	100	at first good	25	pyramidal raising after 4th freezing trial; splitting begins after 7th freezing trial; material becomes tender after 11th freezing trial; material tender, scaling off, splitting after 14th freezing trial
48	P 2	Perfectly intact, clear ring	20	good	—	unaltered after 25th freezing trial
49	P 3	» » » »	15	»	—	» » » » »
50	P 4	In the act of flaking, edges soft at places, ring varies	19	doubtful	59	splitting begins after 1st freezing trial; total splitting after 2nd freezing trial; considerable scaling off, material tender and soft after 14th freezing trial

*) According to expert's opinion the material is inferior.

No. of Sample	Marking of Sample	State of Sample at delivery	Age in years of the material	Preservation hitherto of the material	Loss in weight per cent	Behaviour during the combined Weatherproof-Test
51	P 5	Perfectly intact clear ring	18	good	—	unaltered after 25th freezing trial
52	P 6	» » » »	15	»	—	» » » » »
53	P 7	» » » »	?	»	—	» » » » »
54	P 8	» » » »	37	»	9	pyramidal raising after 6th freezing trial; splitting begins after 7th freezing trial; cleaving, material tender after 25th freezing trial
55	P 9	» » » »	37	»	—	unaltered after 25th freezing trial
56	P10	» » » »	41	»	—	» » » » »
57	P11	» » » »	37	»	—	» » » » »
58	P12	» » » »	37	»	—	» » » » »
		Sent in by: Alb. Bauert, Master-roofer, Zürich.				
59	Q 1	Perfectly intact, clear ring	0	unknown	—	surface very tender after 5th freezing trial

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EXAMINATION AND EVALUATION OF THE RESOLUTIONS OF THE CONFERENCES OF 1884—1893 CONCERNING THE ADHESIVE STRENGTH OF HYDRAULIC CEMENTS

REPORT BY R. FERET.
CHEF DU LABORATOIRE DES PONTS
ET CHAUSSÉES À BOULOGNE S./M.
(TRANSLATED FROM THE FRENCH ORIGINAL.)

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Examination and evaluation the resolutions of the Conferences of 1884—1893 concerning the adhesive strength of hydraulic cements.

Report by **R. Feret.**
Chef du Laboratoire des Ponts et Chaussées à Boulogne s./M.
Translated from the French Original.

A. Resolutions of the Conferences with regard to former methods of testing.

As a result of the Conferences at Munich (1884) and at Dresden (1896), the following proposals relative to the determination of the adhesion of mortars were referred to the new permanent Committee for consideration and report.

„The adhesive force of hydraulic cements as well as of all mortars should be determined by means of standard German apparatus, as used for tension-tests. The test-pieces should be rammed against prisms of ground glass in box-shaped moulds. The test-surface should be 5×5 cm $= 25$ cm^2. The shackle should be capable of easy manipulation so as to ensure a perfectly regular pull.

For all hydraulic cements it is absolutely indispensable that the test-pieces should be always kept in a place saturated with moisture, or in water as is prescribed for tension-test-pieces.

For the purpose of comparing exactly the adhesive force of hydraulic cements, any secondary action on the material to which it is to be fixed must be absolutely avoided. Thus, with all calcareous

cements and especially with all hydraulic limes and all cements, bricks must on no account be used, as they might form a puzzuolana with the lime.

For similar reasons, materials must be selected which are able to resist the continuous action both of acids and of alkalies. Hence the only bodies which may be used in practice are plates of ground glass or of biscuit porcelain, preferably the former. This can be obtained at a small cost, and is only very slightly attacked by lime.

All the glass plates must be ground to exactly the same extent."

At the Conference at Berlin (1890), these proposals were again referred to the permanent Committee for further consideration; the question came before the next Conference and also before the successive meetings of the International Association, but no report has been presented.

Finally, Mr. F. M. Meyer, President of the Committee, a body which seems never to have been properly constituted, resigned on account of his health, and the council decided that the question of determining the adhesive properties of mortars should be studied by a single reporter.

Having been entrusted with the work, after Professor J. Maluga of St. Petersburg had declined it, the writer of the present report thinks that the prolonged state of stagnation in which this question has remained, is principally attributable to the following three causes, whilst most of the other tests of hydraulic cements have undergone and are continuously undergoing improvements:

1. The difficulty of obtaining an «adhesion body»[1]) of uniform structure and at the same time not subject to attack by the various chemical agents which may exist in the materials.

2. The inconsistent results obtained by experimentalists who have worked on the problem outside the International Association.

3. The small importance which has been until quite lately attached to comparing the adhesive qualities of mortars. Their cohesion seems to have been thought the only point worthy of attention.

[1]) The body to which the adhesive properties of the cement are to be tested.

The considerable deve'opment which has taken place of late in armoured cement for building purposes has increased the interest in adhesion tests. We shall see later on, the inference to be drawn from the inconsistency of the results supplied. The difficulties which may be met with in selecting a standard adhesion body ought not to be an obstacle to the adoption of methods of testing which, while we are waitng for such a standard substance, may nevertheless serve in studying in a uniform manner the adhesion of different mortars to different materials.

A method adopted about seventy or eighty years ago for testing a hydraulic cement, consisted in fixing a brick to a vertical wall with the cement. After a certain interval of time, a second brick was fixed with the cement to the first one, then a third to the second, and so on, till the first joint broke. The number of bricks was then taken as a measure of the adhesion of the cement.

Later the method was simplified by fastening two bricks or two stones together with the cement. One of them was then firmly fixed with the other below it. From the lower brick was suspended a scale pan, in which weights were placed till the lower brick was torn off. Then the ratio of the breaking load to the area of the joint, measured the adhesion.

These methods of testing were crude, because the results depended to a large extent on the nature of the bricks or stones which were used, and the test has gradually been improved and made more precise, whilst at the same time the attempt to discover a standard adhesion body has never been given up.

The principal methods which have been adopted have been described by M. Candlot in a report to the French commission for the unification of the methods of testing building materials, and we refer the reader for details to vol. 4, p. 281 of the report which was published by J. Rothschild, Paris, in 1895.

First of all Dr. W. Michaelis placed a small plate of earthenware in the middle of an ordinary briquette mould, of the shape of the figure eight, and the two halves were then filled with the mortar to be tested. The mortar adhering to the earthenware formed a briquette which could afterwards be broken by tension-test in the

usual way. This method did not give satisfactory results and was abandoned even by its inventor.

Dr. Michaelis then proposed another device which found favour for a few years. The mortar to be tested was spread on a plate of ground glass 10 cm by 5 cm, and a second plate of glass was fixed on the top of the mortar so as to form a cross with the first one; the mortar was rammed by means of a special appliance in such a manner that it was always 3 cm thick. After hardening the mortar under water, the adhesion was tested in the testing machine by tearing the plates apart, the glass being held by special clips. One of the chief difficulties of this method, as M. Candlot remarks in his report, was that the glass acted on the lime like a puzzuolana; moreover, the excess of water contained in the mortar, having no means of escaping on account of the impermeability of the glass, formed an insulating layer on one of the glass surfaces, preventing regular and uniform adhesion. This method gave therefore very inconsistent values of the resistance and was soon entirely given up.

Another process which has been applied in various ways consisted in making the «adhesion body» in the form of a small die, on which the mortar to be tested was moulded into a sort of funnel, so that on the removal of the mould it presented a shoulder by which the clips of the testing machine could seize it, while the adhesion body was notched and clasped by a special clip.

In Switzerland, blocks of marble have been used as adhesion bodies, but their preparation was expensive, and it was impossible to obtain absolutely identical surfaces. Dr. Michaelis replaced the marble blocks by blocks of mortar which had been rammed into special moulds, and which was composed of two parts of pure sand and one part of good Portland cement, which latter had been passed through a sieve of 900 meshes to the cm^2. For the purpose of testing the adhesion of a mortar to another substance, such as brick, marble, etc, a block of similar dimensions as the block of mortar was made of the substance. The process was improved by Dr. Tomei who discovered that it was injudicious to ram the mortar into the mould, as the ramming caused a partial separation of the cement and the sand, and thus wrong results were obtained. He preferred to use a mortar which was

gauged to the consistency of a paste, and poured into the mould without ramming. Dr. Michaelis finally adopted the same view.

M. Candlot, in his turn, devised an improvement, which consisted in giving the adhesion body as well as the mortar moulded upon it, the form of half briquettes, so that the two put together made a symmetrical test-piece, with a double shoulder, and could easily be seized by the usual clips of the testing machine. Like Dr. Michaelis, he selected as adhesion body a rammed mortar consisting of two parts of sand and one part of cement by weight. He made the normal mortar to be tested of the same proportions and to the consistency of a paste, and recommended that no excess of water should be used in gauging. By a special design of the moulds, the adhesion body could be terminated at the joint by a plate of any desired material, so that the adhesions of the same mortar to various substances could be compared. The methods adopted by M. Candlot have been recommended by the French unification commission except that some of those, which are founded on a relatively small number of trials, shall not be considered as final.

In fact, the adoption of this method does not seem to have become more general than any of the preceding ones, and the few experimentists, who have since published their results, have generally adopted methods of their own and particularly suitable to the exact nature of their researches. Thus, in a communication to the Congress of Budapest, the writer of the present report described three different methods which he adopted successively.

In any case the rules formulated by the Congresses of Munich and Dresden are long ago out of date. The principal ones, such as the adoption of ground glass and the ramming of the mortar to be tested, have proved faulty in practice. The best thing to do is to make a clean sweep of these antiquated methods, and to formulate a rational system of rules on entirely new principles with regard to methods of adhesion-tests.

In spite of the terms of the question submitted to him, the writer does not consider his task completed by the conclusion just mentioned. In agreement with the late President of the International Association, the author thought that he himself ought to make

some proposals of his own, in reference to the methods which he has to study. In the following, he will develop some of his ideas, which he submits to the International Association for discussion.

B. Study of general methods.

When the joint of two adhering bodies is a plane surface, it is taken for granted that the force required to separate them, varies according to its direction, and that it depends particularly as to whether it is exerted in a direction perpendicular or parallel to the plane of contact between the two bodies. This distinction is expressed by the phrases „normal adhesion“ and „tangential adhesion“. An oblique force can always be resolved into a normal and a tangential component, and will thus call simultaneously into action the two kinds of adhesion.

a) Normal adhesion.

Almost all the tests hitherto made have been in reference to the normal adhesion, and the summary given above shows that whenever it has been desired to express this adhesion numerically, tension methods have been employed in measuring the normal force required to separate two plane surfaces of known area.

This method is nevertheless open to various criticisms.

In the first place, if the test is to be exact, the force must be accurately centred and absolutely normal to the plane of the joint. Otherwise a bending component is introduced, and rupture begins at the edge of the joint under a too low whole load.

In the second place, even if these conditions are realised, it is impossible to shape the shoulders of the test-pieces and the clips of the machine in such a manner, that the force will be uniformly distributed over the area of the joint. As in ordinary tension tests, the tension is stronger at the edges of the joint than at its centre, so that the ratio of the whole breaking load to the area of the joint is less than the actual adhesion. Thus we find that for thesame materials the breaking load is not proportional to the area of the joint.

These difficulties would be avoided by a bending-test under such conditions that we knew exactly the forces developed at every part of the joint. The maximum tension developed at right

angles to the plane of the joint by the breaking load would then be a measure of the normal adhesion.

In a solid, which is acted upon by a bending force, any cross-section is generally subjected to a bending moment, which produces at every point of the section a normal stress, as well as to a shearing force, which produces a tangential stress. The resultant of these stresses is very complicated and may be studied in books on the „Theory of Structures".

The problem is simpler in sections where the shearing force is zero, and where therefore the molecular forces are reduced to tensions and compressions normal to the plane of the section.

In the case with which we are concerned, this condition may be realised in the following way.

In (fig. 1) let A be the adhesion-body, which we shall suppose to be rigidly fixed, and bounded by a horizontal plane XX'. Above this surface the mortar B to be tested is moulded so that it adheres to a rectangular surface, of which CD is the projection, and which measures a cm from C to D, and b cm in the direction normal to the plane of the paper. The upper part of the block of mortar is shaped in such a manner that it can be seized by the clip of a lever E, from the end of which is suspended a bucket F, into which lead shot are poured until seperation occurs.

By this contrivance, the bending moment will be constant throughout that part of the mortar which is below the clip, and no forces will be developed in the section CD except such which are normal, and rupture will take place when the intensity of the stress along the straight line projected at C becomes equal to the normal adhesion of the two materials.

Let the joined weight of the bucket and the lead at this moment be Q kg, and let the horizontal distance from the point of suspension of the bucket to the vertical passing through the centre O of the joint be l cm. Let further the total weight of the block of mortar B, the lever E, and the attachments of the bucket be Q' kg, and the horizontal distance from the mass-centre of Q' to the vertical at O be l' cm.

Now, if the two materials can be regarded as being perfectly elastic up to the moment of separation, that is to say, if the stress

(tension, or compression) remains proportional to the positive and negative strains, then we may consider that the surface of separation CD remains a plane, and that the tension which reaches its positive maximum value at C, $CC' = R$ (fig. 2), is equal to the

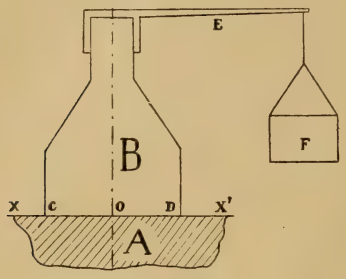

Fig. 1.

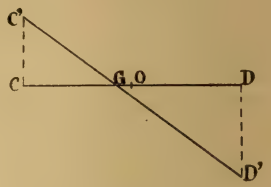

Fig. 2.

adhesion which we desire to measure. The tension decreases uniformly from C to D where it reaches its minimum value (maximum compression), after having changed sign at G. The straight line through point G and normal to the plane of the paper and in which the value of the stress is zero is called the neutral axis of the section CD. Calling the distance OG, d reckoned positive from O towards C we have:

$$\frac{DD'}{CC'} = \frac{GD}{GC} = \frac{\frac{a}{2} + d}{\frac{a}{2} - d}, \text{ hence } DD' = R\,\frac{\frac{a}{2} + d}{\frac{a}{2} - d}.$$

The condition for equilibrium between the external forces and the molecular forces in section CD, is that the algebraical sum of the internal forces in the section must be equal to that of the external forces i. e., equal to $-(Q + Q')$. Now it is easy to see that the sum of the internal forces is the product of the width b of the section and the algebraical sum of the areas of the triangles GCC' and GDD'. We have then the following relation between the unknown quantities R and d.

$$b\,R\,\frac{\frac{a}{2} - d}{2} - b\,R\,\frac{\left(\frac{a}{2} + d\right)^2}{2\left(\frac{a}{2} - d\right)} = -(Q + Q').$$

Now it is also a necessary condition for equilibrium that the sum of the moments of the molecular forces about any axis perpendicular to the plane of the figure, such as the neutral axis, should be equal to the sum of the moments of the external forces about the same axis. This condition is expressed by the following equation:

$$b\mathrm{R}\,\frac{\left(\frac{a}{2}-d\right)^2}{3}+b\mathrm{R}\,\frac{\left(\frac{a}{2}+d\right)^3}{3\left(\frac{a}{2}-d\right)}=(d+l)\,\mathrm{Q}+(d+l')\,\mathrm{Q}'.$$

From these two equations we have:

$$d=\frac{a^2\,(\mathrm{Q}+\mathrm{Q}')}{12\,(l\mathrm{Q}+l'\mathrm{Q}')}$$

and

$$\mathrm{R}=\frac{(6\,l-a)\,\mathrm{Q}+(6\,l'-a)\,\mathrm{Q}'}{ba^2}.$$

Neglecting the variations in the weight of the block of mortar B, assumed symmetrical about a plane passing through the vertical at O and perpendicular to the plane of the paper, we can adjust once for all, by means of a counterpoise, the mass-centre of the lever and its accessories, so that $l'=\frac{a}{6}$. The value of R is then independent of Q' and proportional to the weight Q of the lead and the bucket.

We have aplied the method just described making the two bodies A and B in the shape of prisms with square bases (side 4 cms) and placed end to end. The adhesion body, whatever it may be, is cut or moulded into this shape beforehand. One of its ends is smoothed down with emery. It is then thoroughly soaked in water, drained for a few seconds, and placed horizontally so that the smoothed base of the prism forms one end of the mould in which the mortarprism is made (fig. 3). The mortar consequently adheres to it, and is allowed to set in an atmosphere saturated with moisture. The mould is carefully removed when the mortar has set sufficiently. To facilitate the removal from the mould, and to prevent accidental separation during the removal, it is a good plan to place a piece of well stretched wet paper between the mouldand the back-plate.

Fig. 3.

It is evidently unnecessary that the first half of the prism should consist entirely of the substance whose adhesion we are to determine. It is sufficient if the part near the surface of adhesion be composed of that substance, so long as it is so firmly united to the rest of the prism — which for example may be composed of cement only — that separation will not occur except at the desired place. Thus in the various trials we have made for determining the adhesion of mortars to iron, we have used adhesion bodies made up as shown in fig. 4.

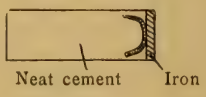

Fig. 4.

In carrying out the adhesion tests, we have adopted the arrangement shown in fig. 5, which is very convenient and which we also use for ordinary bending tests. The opening of the two clips is such that the prism assumes a vertical position auto-

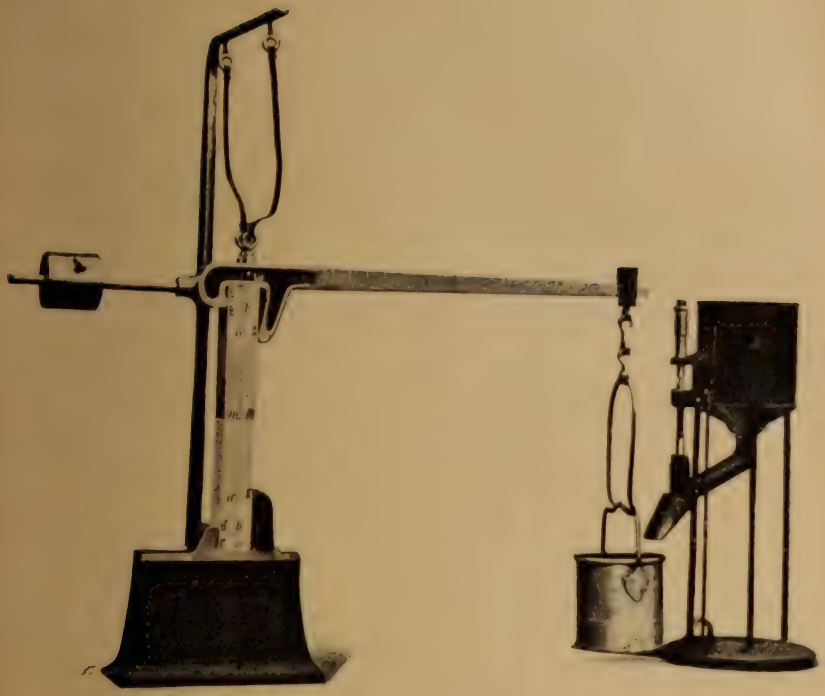

Fig. 5.

matically and the lever a horizontal position. The bending moment is then constant and the shearing force is zero throughout the part of the prism situated between the points at which the two clips bear against the prism. Rupture always takes place without the prism undergoing any perceptible deformation, the lengths l and l' remain therefore unchanged during the experiment and the formula holds good.

As a is 4 cm, Q' will be eliminated if we adjust the counterpoise in such a manner, that the joined mass-centre of the lever, the suspension head of the bucket and the upper half of the prism, shall be $\frac{4}{6}$ of a centimetre from the vertical axis of the prism, and on the same side as the bucket. For the purpose of this adjustment the lever is provided with two pointed rests P, lying in a line at right angles to its length. The bucket having been removed, the other masses are now adjusted so as to balance when the lever is resting on point P in a horizontal position.

A small plane which can pass freely through a hole in the lever serves then as a support for the points P. The distance (l_1) between the points P and the vertical axis of the prism is determined by the formula:

$$l_1 \times Q_1 = l' (Q_1 + q_1) = \tfrac{2}{3} (Q_1 + q_1)$$

in which q_1 denotes the weight of the semi-prism of mortar (about half a kg for a length of 16 cm) and Q_1 that of the lever together with the parts for the suspension of the bucket ($Q' = Q_1 + q_1$).

The horizontal length of the lever, reckoned from the axis of the prism to the point of suspension of the bucket, has been fixed at 54 cm, so that R is always equal to $\frac{6 \times 54 - 4}{4^3} Q = 5\,Q$.

The width of the clips is exactly 4 cm, the same as that of the prism, which latter can therefore easily be centred.

Straps, which must be slack during the test, connect the lever to a fixed bracket, and thus hold it after the rupture of the prism has taken place. Plumb lines may be suspended to the right and left of the bracket, for the purpose of ascertaining at a glance whether the prism is vertical in two different planes.

As in other resistance-tests, it is important that the rate at which the shots are poured into the bucket should be uniform and fixed, because the breaking-load depends upon the duration of the action. We have adopted a rate of 100 grammes per second, the same as already fixed for standard tension-tests, whereby the maximum tensile stress in the joint will be increased at a rate of 500 grs. per cm^2 per second.

The vertical distances $a\,b$, $a'\,b'$, of the two bearings of the same clip are of very little importance; but they should not be too small, as the compressive forces exerted on the prism at the point A, B, A' and B' might be considerable and cause shearing at one of these points, especially if at the same time, the radius of curvature of the corresponding element of the clip is small. In the apparatus we use, this radius is about one cm at the four bearings, and the distances $a\,b$ and $a'\,b'$ are rather

more than 4 cm. Under these conditions we may even in bending-tests of soft mortars, apply the extreme bearing-points B, B', of the clips at only 2—3 mm from the ends C' and C'' of the prism without rupture occurring at the points of contact. Nevertheless, it is better to keep a somewhat greater margin, say 1—2 cm.

We have made various bending as well as adhesion-tests, in order to investigate the distribution, in a cross-section of the prism, of the compressing forces exerted directly against the ends of the prism by the bottom surface of the clips. With this object we have each time tested a large number of similar prisms, in some cases avoiding all contact between the ends of the prism and the clips and in other cases letting the clips bear against the whole end-surface (as in fig. 5). Finally we have also interposed small metal bars, in order to concentrate the vertical pressure to a narrow region either in the neighbourhood of the stretched face, or in that of the compressed face, or midway between the two.

The mean resistances obtained in each way with several series of similar prisms, as well as the means of the differences between the mean resistances and the individual resistances in each case, have been practically the same. We may therefore conclude that it does not matter whether the ends of the prisms abut against the clips or not, and that there is no difficulty in working with prisms with ends cut irregularly. When the ends are cut straight, it is most convenient to let them bear against the bottom of the clips.

A more difficult question to settle is the length to be given to the prism. Theoretically this length may be anything, and the bending moment required to produce rupture ought to be the same, for any distance $A\,A'$ between the bearings of the clips. But in reality secondary molecular forces are developed in the vicinity of the bearing surfaces; these forces vary inversely as the lengths $a\,b$, $a'\,b'$ and the radius of curvature of the clip at the

point of contact with the prism. If the joint is too near the bearing-surfaces, the influence of these secondary actions makes itself felt and modifies the distribution of the stresses, so that the bending moment, which produces a maximum tensile stress equal to the adhesion between the two materials, no longer.

These considerations are confirmed by the following experiments in which each figure is the mean of several trials carried out with similar prisms all made at the same time, and under conditions as nearly as possible identical. For each series of prisms we have calculated the differences between the individual adhesions and their mean values, also the mean of these differences, and finally also the ratio between the mean of the differences and the mean adhesions. We have thus obtained „the mean values of the relative differences", which are the smaller, the better the experiments agree with each other. All the figures on the same line refer to prisms made with the same adhesion body and with the same kind of mortar, and differing only in length.

Different lines refer to mortars of different composition.

	Mean adhesion per cm²		Adhesion of prisms of 15 cm compared with that of prisms of 30 cm	Mean of the relative differences	
Total length CC' of the prisms	30 cm	15 cm		30 cm	15 cm
Distance between joint and the nearest bearing $AM = A'M$	9 cm	1 cm		9 cm	1 cm
	kg	kg	p. 100	p. 100	p. 100
1st test	12·6	10·8	86	7·6	9·5
2nd test	16·5	15·6	95	6·2	11·7
3rd test	20·8	16·0	77	10·3	9·9
4th test	23·0	17·6	77	7·1	9·8
Mean .			84	7·8	10·2

We see that when the distance between the joint and the nearest bearing was only one cm, the resistances have always been found to be less than when the distance was nine cm, and that in the latter case, the results have generally been more concordant.

Other tests, Nos. 5—11 in the following table, were made on the same principle, with lengths between wider limits.

Total length (CC') of the prism in cm		50	32	24	16	14
Distance of the joint from the nearest bearing	$MA = AM'$ in cm	18·5	10	6	2·5	1
Mean adhesion in kg/cm²	5th test	8·6	9·3	7·5	7·4	8·0
	6th test	10·7	9·5	8·1	12·0	7·5
	7th test	8·4	12·5	12·15	10·55	9·9
	8th test	11·5	13·7	16·4	13·5	14·8
	9th test	15·1	19·9	22·0	18·4	15·8
	10th test	19·9	21·1	19·4	21·3	22.25
	11th test	21·9	22·9	19·8	21·2	22·9
Adhesion in per cent of the values in the second column taken as 100	5th test	92	100	81	80	86
	6th test	113	100	85	126	79
	7th test	67	100	97	84	79
	8th test	84	100	120	99	108
	9th test	76	100	110	92	79
	10th test	94	100	92	101	105
	11th test	96	100	86	93	100
	Average . .	89	100	96	96	91
Average of the relative differences (percentages of the mean adhesions)	5th test	14·6	16·7	12·5	17·1	9·9
	6th test	18·1	13·4	6·8	12·3	11·9
	7th test	3·6	4·2	7·7	9·4	9·8
	8th test	12·6	11·4	8·8	8·5	10·1
	9th test	18·4	12·4	8·7	8·7	12·8
	10th test	8·8	11·2	10·0	16·8	12·9
	11th test	6·9	11·4	12·5	16·7	16·4
	Average . .	11·9	11·5	9·6	12·8	12·0

In spite of the inconsistency of the values obtained it seems that all things being the same in other respects, the adhesion increases with the distance between the joint and the nearest bearing up to a certain limit which is about 10 cm and that it then decreases when the prism becomes longer. The consistency of the results, which vary from one series to another, does not in this case appear to be connected by any distinct law of the distance between the bearings A and A'.

In view of these results, we might adopt a total length of 32 cm for the prisms, with a distance of 10 cm between the joint and each of the nearest bearings. This permits of using after fracture the other semi-prism, consisting of the mortar under test, and being 16 cm long, for a bending test, and then for two conpression tests.[1])

Then, taking into account the observations already made, the principal dimensions in fig. 5 would be:

Transverse section of the prisms	4×4 cm.
Length of the semi-prism constituting the adhesion body .	16 „
Length of the semi-prism constituting the mortar under test	16 „
Length of the whole prism	32 „
Distance from the joint to the inner bearings of the clips . . . . . $A'M = MA =$	10 „
Vertical distance of the bearings of the same clip . . . . . $ab = a'b' =$	4 „
Distance from the outer bearing of each clip to the bottom of the clip where the prism touches it $BC = B'C' =$	2 „

Although the second column of the previous table gives the mean-value of the relative differences obtained in the various series of tests which were made under the conditions just defined, it was necessary to repeat the experiments with a larger number of prisms, in order to form a better idea of the accuracy of the method.

The first set of these tests was made with 30 similar prisms; the following were the principal characteristics:

Adhesion body. Mortar: 1 part of fine sand and 1 of cement; this mortar, moulded into prisms measuring $16 \times 4 \times 4$ cm, had been kept for nine years in water. The surface of adhesion was smoothed some hours before moulding the test-mortar on to it.

Test mortar. Mortar of plastic consistency made of one part of Portland cement and three parts by weight of a rather coarse shelly sand.

[1]) This method is justified by simultaneous researches in which we have studied bending-tests made with constant moment.

The prisms were kept a week in damp air, and then for five weeks in water at 15 deg. C. before being tested.

Rate of increase of test load. 100 grs. per second.

Time taken for 30 breakages with one operator: 50 minutes.

Mean of the 30 adhesions: 11·49 kg per cm^2.

Mean of the absolute values of differences 1·10 kg per cm^2 or 9·5% of the 11·49 kg.

Sum of the squares of the differences: 57·20.

Mean difference $= \sqrt{\frac{57{\cdot}20}{29}} = 1{\cdot}4$ kg/cm^2 = 12·2 per cent of 11·49

Probable difference 0.94 kg per cm^2 = 8·2 per cent of 11·49.

In a second series of tests we proposed to compare three cements of the same origin, but differing slightly in composition. Ten prisms were made of each of the three cements. They were first tested for adhesion and then their halves were tested for bending (10 experiments with each cement, with a distance AA' of 4 cm), and then for compression (20 experiments with each cement). The data and results were as follows:

Adhesion body. Mortar: 1 part of cement to 2 parts of the same fine sand; kept for nine years as above mentioned and treated in the same manner.

Test-mortars. Plastic mortars of 1 part of Portland cement with three parts by weight of a rather fine yard sands gauged with 14 per cent of water.

The prisms were kept a week in moist air, and then three weeks in water at 15 deg. C. before being tested.

Rate of increase of test-load in the two series: 190 grammes per second.

Time taken for 30 ruptures in adhesion-tests with a single operator: 38 minutes.

Time taken for 30 ruptures in bending-tests with a single operator: 45 minutes.

			1st cement	2nd cement	3rd cement
Mean of ten adhesions:		kg/cm^2	14·03	15·30	13·04
Mean of the absolute values of the differences	absolute	kg/cm^2	1·45	0·56	2·18
	relative	p. 100	10·3	3·7	16·6
Mean of the ten resistances to bending:		kg/cm^2	24·84	25·11	25·49
Mean of the absolute values of the differences	absolute	kg/cm^2	0·82	0·95	0·85
	relative	p. 100	3·3	3·8	3·3
Mean of the 20 resistances to compression:		kg/cm^2	103·4	98·4	103·2
Mean of the absolute values of the differences	absolute	kg/cm^2	4·90	4·38	3·71
	relative	p. 100	4·7	4·4	3·6

We will now quote among other adhesion tests made with various mortars[1]), a third set carried out on five plastic mortars of different compositions made with the same cement, using six prisms of each mortar.

Adhesion body. Mortar 1 to 1, of rather fine building sand, prepared and kept like those in the preceding trials.

The prisms were kept one week in a damp atmosphere, and then 3 weeks in water at 15 deg. C.

Rate of increase of test-load: 100 grammes per second.

Time taken for 29 ruptures with a single operator: 75 minutes.

Test-mortars made with the same cement	Nature of the sand	No sand used	very fine sand		very coarse sand	
	Weight of sand to one of cement	0	2	5	2	5
	Weight of water used for gauging 100 parts of dry mixture	24·1	16·7	17·7	10·8	7·0
	Weight in gr. per liter of fresh mortar	2158	2006	1923	2325	2257
Adhesion in kg/cm² — Individual results		21·6	12·2	—[2])	26·3	3·2
		25·5	13·4	6·1	29·7	4·0
		25·8	14·6	6·2	30·4	5·8
		28·2	15·2	6·5	32·5	6·6
		34·7	15·5	6·7	32·7	9·4
		42·4	15·9	6·7	38·8	10·6
Adhesion in kg/cm² — Average		29·7	14·5	6·4	31·7	6·6
Means of the absolute values of the differences	absolute: kg/cm²	5·90	0·55	0·24	2·93	2·27
	relative: per 100	19·9	3·8	3·7	9·2	34·4

This trial is particularly interesting, as it shows how much the regularity of the results from one series of prisms may vary, according to the composition of the mortar. Whilst the mean of the relative differences was only 3·8—3·7 per cent with fine sand mortars, it went up to 34·4 per cent with the poor mortar of coarse sand. The differences therefore are not due to any fault in the method of testing, but to real differences in the adhesion of the mortar according to the distribution of sand and cement at the surface of the joint.

[1]) These tests with mortar of fat lime and puzzuolana are described in another report on the subject of Problem 11 (Puzzuolana tests).

[2]) One prism was accidentally broken in removing it from the mould.

The face of the mortar which remains free during moulding, is always rather less regular than the faces which are in contact with the bottom or sides of the mould. We must therefore avoid having this face in contact with the clips in both adhesion and bending tests.

On the other hand, we have shown that when the prisms are dipped in such a manner that the lateral faces which were vertical when in the mould, are placed horizontally in the bath, the uppermost face will harden a little more than the other. Hence in both, adhesion as well as in bending-tests, we obtain somewhat greater or smaller resistances, according to whether the face is subjected to tension or compression by the manner in which the prism is fixed in the clips during the test. The consistency of the results are practically the same in the two cases.

In the table below, the figures in the upper line are the mean adhesions of prisms tested with the faces in tension which were uppermost in the water, and the figures in the second line correspond to similar prisms in which the face was subjected to compression. The figures in the third line are the differences between the resistances in the two cases. In the fourth line, the second set of resistances are expressed in percentages of the first set. Each column refers to a series of prisms differing from the others, either in the adhesion body, or in the test mortar, and generally in both.

Upper face in tension:	$R_1 =$	11·2	11·3	12·5	13·0	15·2	15·9	16·3	17·4	18·1	18·2	18·2	19·3	23·4 Averages
Upper face in compression:	$R_2 =$	11·7	10·2	12·7	12·4	14·4	14·4	15·6	16·4	17·0	19·0	15·9	22·1	22·6
	$R_1 - R_2 =$	-0·5	1·1	-0·2	0·6	0·8	1·5	0·7	1·0	1·1	-0·8	2·3	-2·8	0·8 + 0·43
	$\frac{100\,R_2}{R_1} =$	104	90	102	95	95	91	96	94	94	104	87	114	95 97

Although the mean of the absolute differences is very small and the mean of the relative differences is nearly 100, still the numerical predominance of the values of R_1 over those of R_2 justifies the observation just made. To avoid this source of error in the tests, it should be a rule that the lateral faces which were vertical during moulding, and against which the clips will bear, should also be vertical in the water so that they may harden equally.

The contrivance adopted in the case which has just been studied is based on the same principle as that represented in fig. 1, but differs from it in several details.

In fig. 1, only one of the faces of the adhesion body need be dressed, while in fig. 5 the body must be moulded or cut into prisms of accurate size, and this, in spite of the simplicity of the shape, renders it more difficult to make.

Also in the case of materials, to which the mortar adheres badly, the former arrangement has the additional advantage over the latter, that the surface of adhesion may be taken as large as we please, while in the case shown in fig. 5, there is a practical limit to the sectional area of the prisms.

On the other hand, in the double-prism method, the shape of the test-mortar is simpler and more easily produced than in the case of fig. 1, where the comparatively large size of the joint requires the mortar block to be made thinner upwards, so as to give the lever a better hold. It could however be made in a prismatic form, if a square iron rod with a cross-section of say 4×4 cm, and co-axial with the prism was fixed in the mass of the block and seized by the lever; not only would this rod have to be perfectly centred however, but its faces would have to be exactly parallel to the sides of the joint. There would also be reason to fear that, in the heterogeneous mass it would form with the mortar, the molecular actions would not be transmitted, in the same manner as in the homogeneous mass, so that the distribution of the stresses in the joint might be quite different from that indicated by calculation.

Finally, the two contrivances present this important difference, that in the first one, the mortar is applied to an adhesion body placed horizontally, while in the second one, the joint is vertical during moulding. This condition is evidently more favourable to the formation of a good joint and in getting rid of any accumulated air bubbles. Besides, in the first case, with some soft mortars, a partial separation of sand and cement may occur, which would make the composition at the joint different to other parts of the mortar.

Hence we have given the preference to the system of double prisms, and have specially studied, as shown above, the conditions under which it may be suitably applied.

b) Tangential adhesion.

Very few tests on tangential adhesion have been made, and its determination is a very difficult one.

The first method which occurs to anybody is, to apply on each of two blocks an equal pressure, but opposite in direction and parallel to the plane surface of contact, (fig. 6). But as the forces in reality cannot be confined to the plane of contact, normal components must always exist. Besides, the distribution of the molecular stresses in the plane of contact is unknown, and depends on the manner in which the external forces themselves are distributed over the surfaces they act upon. The state of things would remain the same, if instead of applying pressures, the two blocks, or one of them, were subjected to tension, say by means of hooks fixed in the mass of the blocks.

Another method which is especially applicable for making determination of the adhesion of mortar to iron, consists in embedding an iron rod in the mortar and measuring the pull or pressure needed to move it in the mortar (fig. 7). This method is open to the same objection as the last, as regards the uncertainty about the distribution of the forces over the surfaces of contact between the mortar and the iron. Besides, the changes in volume, which the cement is subjected to in setting, introduce disturbing forces by tightening or relaxing the grip on the rod.

Fig. 6.

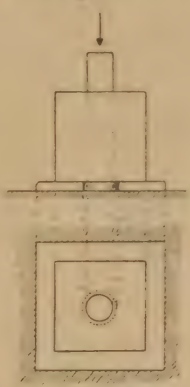

Fig. 7

Hence neither of these methods affords an absolute measure of the tangential adhesion. The most that can be done is to define all the details of the experiment so exactly that comparative results can be obtained from similar trials carried out with different materials. The results which are obtained are never more than approximate values of the adhesion which it is desired to measure.

No doubt more exact figures would be obtained if the surface of contact were a circle, and if then one of the materials were firmly fixed, while the other was subjected to a couple whose axis passes through the centre of the circle, and is normal to the surface of contact. If every disturbing influence could be eliminated, and one could be sure that the shearing stresses continued to be proportional to the strains, then the intensity of the tangential adhesion would be measured by $\frac{2\,M}{\pi\,r^3}$, where r denotes the radius of the circle and M the moment of the twisting couple which produces the separation.

During the trials of the Official French Committee on Armoured Cement, M. Mesnager, at the Ecole des Ponts et Chaussées, designed a contrivance with the object of making determinations of the tangential adhesion between two substances by measuring their mutual slipping before separation.

The principle adopted in this method is shown in fig. 8, where the load P is extended in the plane of the joint by the aid

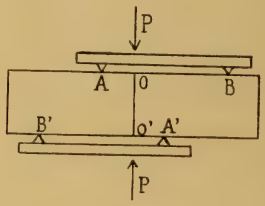

Fig. 8.

of four props A, A', B and B' placed so that $OB = O'B' = 2\,OA = 2\,O'A'$. Under these conditions the tangential load acts exactly in the plane of adhesion and its magnitude is $\frac{P}{3}$.

To avoid the risk of breaking the materials in the line of the props, metallic plates may be placed between the props and the test-piece and thus distribute the pressure without modifying the magnitude of the separating force exerted at the joint (fig. 9).

This method is very attractive becourse tests can be made on tangential adhesion with double prisms identical with those adopted for trials on normal adhesion. We have, therefore, also made tests with the object of verifying the method.

For this purpose, and also for generalising the problem, we have made tests to find the magnitudes of the separating load for different values of the ratio of OA to OB.

Let a be the lengths of OA and $O'A'$ and $b > a$ the lengths of OB and $O'B'$.

The equal reactions at A and A' and those at B and B' are measured by $\frac{b}{b+a} P$ and $\frac{a}{b+a} P$ respectively; and it is easily seen that in a plane parallel to and at a distance x from the joint, the shearing load is constant and equal to $\frac{b-a}{b+a} P$ as long as x is smaller than a, i. e. between A and A', and that it is constant and equal to $-\frac{a}{b+a} P$ for values of x taken between a and b, i. e. in the regions $B'A$ and $A'B$. The bending moment, which is zero at the plane of adhesion, is measured by $\frac{b-a}{b+a} Px$ in the first interval and by $-\frac{a}{b-a} P\ (b-x)$ in the two other intervals. These values are graphically shown in fig. 10, where the bending moments

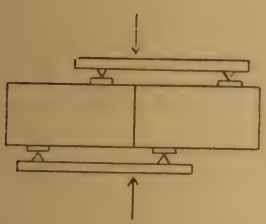

Fig. 9.

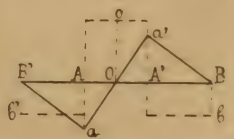

Fig. 10.

are represented by the ordinates to the full lines, and the shearing loads by the ordinates to the dotted lines. $B'b' = Bb = -\frac{a}{b+a} P$, $Oo = \frac{b-a}{b+a} P$ and $-Aa = A'a' = \frac{b-a}{b+a} aP$.

Hence, whatever may be the values of a and b, the bending moment is zero at the section of the joint and the total shearing stress is equal to $\frac{b-a}{b+a} P$, which depends only on the relative values of b and a.

If the stresses, which are actually produced, agree with those obtained by calculation, and if the joining together of the same two materials be identically the same, then the separation should take place — with different values of a and b — under such loads for which the shearing forces, as deduced from above formula, have the same values.

We have made two series of trials, using prisms with a square base of 4 cm side and joined end to end. Iron wires of 2 mm diameter were used as props; in several trials the wires were separated from the prisms, fig. 9, by plates of iron 9 mm wide and 1 mm thick; in another trial the diameter of the wire was 5 mm.

The adhesion bodies were made of two hard mortars of different compositions, and as test-mortar a plastic mortar was adopted containing one part of Portland cement to two parts of natural sand. The double prisms were kept in soft water and broken after 75 days in the first series of trials and after 59 days in the second series.

The adjacent table shows the conditions under which the trials were made and gives also the results obtained.

Values of a and b in cms. / Value of $\frac{b-a}{b+a}$			1:2 $\frac{1}{3}$	2:4 $\frac{1}{3}$	(2:4)[1] $\frac{1}{3}$	3:6 $\frac{1}{3}$	(3:6)[2] $\frac{1}{3}$	1:3 $\frac{1}{2}$	3:9 $\frac{1}{2}$	1:4 $\frac{3}{5}$	2:8 $\frac{3}{5}$	1:9 $\frac{4}{5}$
First series of prisms	Values found for P (in kgs.)	Individual resistances	435 528 514 510 562[3]) 440[3]) 647[3])	540 582[5]) 634[5]) 663[5])	690 659 553 778	439[4]) 393[4]) 441[4]) 385[4]) 412[4])	380[4]) 402[4]) 423[4]) 394[4])	435 420 489 455 353 323 352		352 427 324 411[3]) 425[3]) 426[3])		347 294 340 350 396
		Mean	519	605	670	414	400	404		394		345
	Mean of the absolute values of the relative differences		9·8	7·2	9·5	5·0	3·2	13·0		9·4		6·6
	Shearing force (calculated)		173	—	223	—	—	202		236		276
	Proportional figures		100		129			117		136		160
	Bending moment (calculated)		—		—	414	?	—		—		—
Second series of prisms	Values found for P (in kgs.)	Individual resistances	367 405 434 376 481 448[3]) 485[4]) 429[4])	513 529 400 480[3]) 453[5])	542 529 544 408[3])	395[4]) 401[4]) 424[4]) 418[4]) 441[4]) 370[4])	392[4]) 339[4]) 385[4]) 322[4])	323 459 340 368 351 355	244[4]) 221[4]) 229[4]) 206[4]) 235[4])	265 251 270 238 259 309 308[3])	317[4]) 309[4]) 305[4]) 338[4]) 310[4])	312 265 291 250 265
		Mean	428	475	506	408	360	366	227	271	316	277
	Mean of the absolute values of the relative differences		8·0	8·2	9·6	4·9	8·0	8·6	4·8	7·7	3·0	7·2
	Shearing force (calculated)		143	158	169	—	—	183	—	163	—	221
	Proportional figures		100	111	118			128		114		154
	Bending moment (calculated)		—	—	—	408	?	—	340	—	379	—

[1]) Props separated by plates from the prisms of the first series; wires of 5 mm diameter and without plates are used as props for the prisms of second series.

[2]) Props separated by plates from the prisms of the two series.

[3]) Rupture takes place at one of the props A or A' and then joins the plane of adhesion, fig. 11.

[4]) Rupture takes place across one of the prisms along the line of one of the props A or A', fig. 8.

[5]) Rupture across one of the prisms following one of the lines AB' or $A'B$, fig. 8.

It will be seen that, in the case of the largest values of a and b, rupture was produced by bending, across one of the materials before separation took place, and that interposition of metal plates did not prevent it. The mean-values of the moment of rupture show important differences according to the conditions of the trials.

In each series of blocks separated by shearing, there is no notable difference between the obtained values of P when rupture is produced in the plane of the joint only, or when the prism is also split at one of the supports as shown in fig. 11. On the

Fig. 11.

contrary the magnitude of the shearing stress which, according to theory should correspond to the separating load, varies according to the conditions of the trials. It clearly increases, except in rare cases, with the ratio of b to a.

As all the blocks of the same series were similar and gave only small relative differences in each group of trials made under identical conditions, the diversity of the calculated means for the shearing forces tend to prove that the actions which really take place in the plane of the joint do not agree with those estimated by theory.

In short, only few experiments have been made with the object of making exact determinations of the tangential adhesion between two bodies, and none of the adopted methods has given satisfactory results.

The most practical of them all seems to be that which has just been described. It has the advantage of enabling us to operate with double prisms which are prepared in the same manner as those used for determining the normal adhesion, with that difference however, that they are not required to be so long.

If this method be adopted provisionally, the best values to select for a and b will undoubtedly be 1 cm and 3 cm respecti-

vely, as for these values the ruptures, as shown in the two series of trials related above, were always produced by separation only. The total tangential stress developed in this case in the plane of the joint, should be equal to half of the total ultimate load, and according to the results we have obtained the error which we would commit by deducing it from the actual results of the trials would no doubt be small.

But, as we do not know how the shearing force is distributed over the plane of the joint, we cannot yet deduce from its total magnitude the value of the stress, which will be developed at the point where the separation commences, i. e. the tangential adhesion to be searched for.

C. Standard Tests.

Whatever may be the methods adopted for carrying out the normal and tangential adhesion tests, they should admit of varying at will, either the adhesion body or the mortar to be tested, so that one may compare the adhesions of the same mortar to various materials, or the adhesions of different mortars to the same substance.

Besides the form of the blocks should be simple, so that it can easily be defined and produced. The adhesion bodies in particular must be capable of being made of a uniform size from all sorts of materials at moderate cost.

The double-prism method, as above explained, satisfies these conditions and might, we think, be adopted as a standard test method. We should then have the means of comparing the adhesions of any mortar to any substance under identical conditions.

The same method would also serve for testing glues, mastic, etc., and permit of measuring, under the same conditions, their adhesion to various substances which they may be required to join together.

But ought we to stop there? Is it not also advisable to fix upon a standard adhesion body and a standard mortar, by means of which we might classify all the hydraulic cements according to their adhesive power, measured under well defined conditions?

In order that such tests may be of practical interest, we must be sure that whatever may be the nature of the adhesion body, and the composition of the test-mortar, the classification of the binding agents will remain the same; otherwise there would be little advantage in knowing that one cement is found to be more adhesive than another in the standard tests, if that classification may possibly be reversed with the stones and composition used in practice.

The results hitherto obtained seem to show that the adhesion is not a characteristic property of each binding agent, but depends much more on the composition of the mortar and on the nature of the material to which it is applied than upon the nature of the binding material.

We are, therefore, of opinion that adhesion tests, although very useful as practical tests when carried out with materials and proportions as used by every builder, are only of moderate interest as standard tests applied to all cements under precisely the same conditions.

Nevertheless, cases may occur where, failing special indications due to circumstances, the experimentist desires to be guided in his choice of mortars and adhesion bodies by a set of comparative tests.

If it is a question of comparing various mortars with one another, it is necessary to choose, as far as possible, an adhesion body which produces no chemical action with the mortars. We have already seen that this problem presents serious difficulties, and that finally Dr. Michaelis, M. Candlot, and the Commission française des Méthodes d'Essai agreed upon adopting as standard adhesion body a Portland cement mortar, composed preserved and prepared according to a well defined method. We might then provisionally adopt a mortar of this kind, exactly defined in the conclusions given below, except for proving that the adhesion of a given mortar to such a body is independent of the kind of Portland cement used in making the latter, or of the age of the mortar beyond the imposed limit, or of the manner in which the joint has been prepared, etc.

If it is a question of comparing the adhesion of the same mortar to various bodies, then a first condition found necessary by former experimentists is that the mortar should be gauged to

a plastic consistency. When the International Association has defined such a mortar for standard resistance tests, then we shall have to see whether it will also answer for adhesion tests, and to adopt it if suitable for this purpose. In the meantime, we may stipulate that the standard plastic mortar for adhesion tests should consist of one part of cement and three parts (by weight) of the standard sand to be decided on by the special Committee appointed for this purpose. The amount of water to be used for gauging should also be fixed, for it greatly influences the adhesion If the portion of water is insufficient the adhesion is weak and irregular, while an excess of water may cause partial separation of the cement and the sand and thus destroy the homogeneity of the mortar. It is impossible to fix upon a binding material from which we may derive a standard mortar of always exactly uniform nature.

Finally if it should be proved that the fears expressed above are exaggerated, and that the adhesions of the various binding materials, whatever they may be, always follow in the same order, when the nature of the adherence body and the composition of the tests-mortar are simultaneously altered, we would then be able to compare the relative adhesive powers of various binding materials by measuring the adhesions of standard mortars (as already defined) made with these binding agents, to the standard adhesion body, as already defined.

With regard to the manner and time in which the prisms have to be kept before tested, we must conform to the same rules as used in other tests of hydraulic cements. But as the prisms cannot as a rule be removed from the moulds after 24 hours, as they are not yet sufficiently hardened and the adhesion is stil feeble, we ought to extend the exposure to damp air, to seven days before immersion in water.

Conclusions.

A. The principal conclusions arrived at by the Conferences of 1884—1893 with regard to the adhesive force of hydraulic cements have been nullified by experience, and no further notice need be taken of the decisions of the Conferences on this subject.

B. Given two bodies which adhere together at a plane surface, the intensity of the force necessary to pull them apart varies according to whether the force is exerted in a direction normal to the joint or parallel to it. In the first case the resistance is called the normal adhesion, and in the second case the tangential adhesion.

When it is desired to determine either of these, the testpiece must be in two parts, the adhesion body, prepared beforehand, and the test-mortar, moulded in contact with the former, to which it adheres at the joint.

a) Whatever may be the nature of the adhesion body and of the test-mortar, the normal adhesion should be determined as follows:

As far as possible the adhesion body should be made in the form of a rectangular prism measuring 4 × 4 × 16 cm. One of its ends should, a short time before it is applied to the mortar, be dressed and roughened on a plane surface by means of emery which has been passed through a sieve of 900 meshes to the cm^2, but remained as a residue on a sieve of 4·900 meshes to the cm^2. In the case of a substance being chosen as adhesion body which does not lend itself readily to be made into such a prism, we may make most of the prism of any substance which is hard enough and sufficiently adhesive to the adhesion body, say of neat Portland cement, and only make that part of the prism which is near the joint of the adhesion substance.

If the adhesion body is porous, it must be immersed in water till it is completely soaked. It should then be quickly wiped and the test mortar moulded on to it at once.

In all cases the moulding must be done as follows:

On a horizontal polished plate of marble or metal a wet sheet of paper is stretched, on which the adhesion body is placed in a horizontal position. Rectangular plates of metal, previously wiped with a greasy cloth, are clamped on to the adhesion-body so as to form a mould measuring inside 4 × 4 × 16 cm in the continuation of the adhesion prism, and further bounded at one end by the dressed face of the latter.

The mortar to be tested, gauged to a plastic consistency should be poured into the mould with a trowel and pushed with the finger

into all the corners of the mould, particularly at the joint. The free surface is smoothed with the trowel.

A sufficient number of prisms should be made at the same time so as to be able to test at least six after the respective periods of preservation.

The moulds and their contents are to be preserved in an atmosphere saturated with moisture, sheltered from currents of air and from the direct rays of the sun, and kept as far as possible at a temperature of 15 to 18° C.

The prisms, when sufficiently hard, are carefully removed from the moulds and replaced in the same moist atmosphere, from which they are removed after seven days and put into the hardening medium.

If this medium is water, the prisms are to be immersed with the lateral surfaces vertical which were also vertical in the mould. The prisms are to be torn apart by bending at a constant moment, in an apparatus similar to that represented in fig. 5 and fulfilling the following conditions:

The distance between the two branches of the lower clip must be such that the prism is quite vertical when two opposite faces of it are in contact with the clip.

The two clips must be exactly 4 cm wide and must bear upon the prism along four straight lines perpendicular to its length, where the radius of curvature of the bearing surfaces of the clips should be at least one cm.

The vertical distance between the two bearings of the same clip (*a b* and *a' b'* fig. 5) is about 4 cm.

The lever is to be so made that when the prism is vertical and the upper clip is bearing on two opposite faces, the horizontal distance from the point of suspension of the bucket to the vertical axis of the prism is 54 cm.

The lever is to be so balanced that in the position just indicated the common mass-centre of the upper semi-prism, the lever and the suspension of the bucket, which are not weighed with the latter, is on the same side as the bucket, and at two thirds of a cm from the vertical axis of the prism.

The prism is to be set vertically in the clip so that the latter bear on the faces which were vertical in the mould.

The distances from the plane of the joint to the bearings of the clips on the compressed face ($A'M$ and MA in fig. 5) should are 10 cm.

Shot is poured into the bucket, which is suspended at the end of the lever, at a constant rateof 100 grs. per second. The apparatus is to be supplied with a contrivance for stopping the shot when the prism breaks.

If the rupture begins in the plane of the joint, on the face which is in tension, then the number of kg per cm^2 which expresses the normal adhesion between the two substances is obtained by multiplying the total weight (in kg) of the bucket and the shot by 5.

If on the other hand rupture begins either in the adhesion body or in the mortar under test, all that can be said is that the adhesion between the two substances is greater than that product.

b) On account of the few trials which have been made up till now on the tangential adhesion, it is impossible for the present to point out a method by which it can be accurately measured.

It is to be hoped that results may be obtained by means of a contrivance based upon the principle of double prisms, such as we have proposed to use for trials on normal adhesion.

C. a) If we want to compare the normal or tangential adhesion of various mortars to the same substance, we must prepare a standard adhesion body as follows:

One part of artificial Portland cement which has been passed through a sieve of 900 meshes per cm^2 is mixed with twice its weight of a sand, from the same source as the standard sand adopted for cement resistance tests, but consisting solely of grains which can pass through holes of one mm in diameter but not through holes of half a mm in diameter.

The mixture is gauged with 9 per cent of its weight of water, and is well rammed into moulds of the same form as the adhesion body. The blocks are removed from the moulds after 24 hours, and kept in water — for at least 28 days — before they are used. The face to which the test-mortar is to adhere is prepared as explained above.

b) When it is required to compare the normal or tangentia adhesion of the same mortar to various materials, a standard plastic mortar must be used.

Until the International Association has defined such a mortar for ordinary tests on the strength of hydraulic cements, we shall use for adhesion-tests a mortar composed of one part of cement and three parts by weight normal sand.

Until the Association has defined a normal sand, we will use for adhesion-tests a siliceous sand consisting of rounded grains, and prepared by mixing equal weights of three sizes of grains limited by sieves with holes of 2 mm, 1·5 mm, 1 mm and 0·5 mm diameter respectively.

The amount of water used for gauging must be exactly defined, so that whatever binding-agent is used, the mortar is always of the same plastic consistency, without being too soft.

c) If it appears from a sufficient number of new experiments that the adhesions of various cements can always be arranged in the same order, when the nature of the adhesion body and the composition of the test-mortar are varied simultaneously for all, then standard adhesion-tests may be instituted by measuring the normal or tangential adhesion of the standard mortar, as defined in (b), made with the cement in question, to the standard adhesion body defined in (a).

The conditions and the periods of preservation of the tests-pieces will then be the same as for the ordinary standard resistance tests, except that the exposure in the final medium will not take place until after seven days induration, in an atmosphere saturated with moisture, and in the conditions already defined.

D. The methods defined in B may be applied to measure the normal and tangential adhesions of glues and similar materials, only with this difference that the test-mortar must be replaced by a second adhesion block, which may or may not consist of the same substance as the first one, as the case may be.

The two adhesion bodies will be joined with the glue to be tested, taking care that it covers the two faces of the joint completely. After the time allowed for the glue to set has elapsed, the force necessary to break the joint is measured as above directed.

In all cases the report must state exactly the conditions under which the test has been carried out, specially mentioning the nature of the adhesion bodies, the condition of their joined surfaces, the manner of applying the glue, the thickness of the joint, the conditions and time allowed for setting, the state of the surfaces after rupture, etc.

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BRUSSELS CONGRESS 1906.
REPORT ON THE PROBLEM 11.

TO ESTABLISH METHODS FOR TESTING PUZZOLANAS WITH THE OBJECT OF DETERMINING THEIR VALUE FOR MORTARS.

REPORT BY THE CHAIRMAN
OF COMMITTEE 11
G. HERFELDT, ANDERNACH.

(TRANSLATED FROM THE GERMAN ORIGINAL.)

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Report by the Chairman of Committee 11

G. Herfeldt, Andernach.

(Translated from the German Original.)

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Wagner P., Ingenieur, Köln a/Rh., Mechtildisstraße 6.

France:	A. Mesnager, professeur à l'Ecole des ponts et chaussées, ingénieur des ponts et chaussées, Paris, rue de Rivoli 182.
	F. Feret, chef du laboratoire des ponts et chaussées, Boulogne-sur-Mer (Pas de Calais).
Holland:	A. P. M. Kaptein, Abteilungsvorstand bei den niederländischen Staatseisenbahnen, Utrecht.
	Van der Kloes J. A., Lehrer der Baumaterialienkunde an der polytechnischen Hochschule in Delft.
Italy:	A. Arlorio, Tenente Colonnello nel R. Genio Militare di Bologna.
	Canevazzi S., professeur à l'Ecole royale d'application pour les ingénieurs à l'Université, Bologna.
	U. Cattaneo, ingegnere addetto al Laboratorio per le prove dei Materiali da costruzione della Rete Adriatica, Ancona.
	Geradini, professeur de l'Ecole polytechnique, Rome.
	Giambruni, ingénieur, génie civil, Naples.
	G. Giorgis Dr., professeur de chimie, Ecole royale d'application pour les ingénieurs, Rome.
	C. Guidi, ingénieur, professeur de l'Ecole politechnique, Turin.
	O. Rebuffat, Dr. prof. de chimie, Ecole royale des ingénieurs, Naples.
Austria:	A. Greil, Bauinspektor, Vorstand der städtischen Materialprüfungsstation, Wien, I., Rathaus.
	H. Hillinger, Hofrat im k. k. Handelsministerium, Wien.
Russia:	N. Bogdanoff, ingénieur civil, St. Pétersbourg, Wass. Ostr. 4^{me} ligne 47.
	W. Herzenstein, ingénieur civil, St. Pétersbourg, Wossnessensky 31.
United-States:	A. Lundteigen, Chemist, Union City, Mich.

Mr. W. Herzenstein, engineer in St. Petersburg, has left the Committee and his place has been taken by Mr. F. Konossewitch, mechanical engineer of the Committee for road construction in St. Petersburg.

At the International Congress for Testing Materials held at Budapest, the sub-committee for problem 11 instructed Mr. Feret, chief of the laboratory of the ponts et chaussées department in Boulogne, to arrange a programme of work for the purpose of testing the puzzolanas in a uniform manner.

This programme was to be carried out contemporaneously in a number of different laboratories and with the same materials.

Mr. Feret has designed this programme with great particularity. It includes all points which can possibly be of importance for testing the puzzolanas technologically as regards their mortar value, and proposes experiments for ascertaining this exactly. The Committee determined that the test should be carried out with mortar or cement that had been mixed to a plastic consistency so as to include not only experiments with very finely powdered puzzolanas which give the highest values but also tests with coarse grain puzzolanas as employed in buildings particularly in Italy.

This programme of work corresponding in all respects to the conditions proposed by the Committee, has been published by Mr. Feret, both in French and German, in «Baumaterialienkunde», the organ of the International Union for testing materials (year VII, vols. No. 1 to 4, 7, 8, 10, 11 and 13*).

Connected with this extensive work the undersigned requested the respected members of the Committee who wished to take part in the experiment to indicate before the 15th August 1902 the weight of puzzolanas which would be necessary for their experiments. The exchange of materials had to take place by the first of Oct. 1902 at the latest. In order to be able to compare the results with one another it was requested that all the gentlemen who intended to make experiments should have the same sort of lime (fat lime from Liège) sent to them.

*) All members who were not subscribers to «Baumaterialienkunde» had the respective numbers sent to them, either in French or German.

Very little attention has been paid to this request, however, and the undersigned has only sent to Mr. Feret the variety of puzzolanas (tufa) occurring here and Liège lime, and also to Prof. Gary in Gross-Lichterfelde and Messrs. Bogdanoff and Konossewitch in St. Petersburg.

The results which Mr. Feret has obtained constituting an extended work in regard to the whole programme, have been laid before the St. Petersburg Congress in a special report.

Prof. Gary was unable to carry out the programme owing to the removal of the Testing Institute from Charlottenburg to Gross-Lichterfelde, which was especially to be deplored as in this case the experiments would have been conducted by a particularly competent experimenter and well instructed assistants in a laboratory provided with the most complete apparatus for the purpose.

I have not yet received any results form Messrs. Bogdanoff and Konossewitch.

The undersigned has carried out 40 experiments in accordance with the Feret programme in his laboratory with tufa, the results of which are appended as Appendix C.

The whole of the Feret programme was too much for us to carry out in addition to the other necessary work and I may add that, in my opinion, the small amount of co-operation which we have received from the other members is largely due to the extent to which they have been frightened off by the elaborate character of the experiments proposed, such as grinding up the puzzolana till it all went through a 5000 mesh sieve, sifting the lime and preparing sand of a special grain all of which of course takes an enormous amount of time.

For carrying out this work a great deal of time, good apparatus and well trained laboratory assistantes are essential.

The president, councillor von Tetmajer, informed the chairmen of the Committees on the 3rd April of this year that the Committee intended to send the papers and reports so early to the members of the Congress that they would be able to look through them before starting to St. Petersburg. He assumed that otherwise an adequate and instructive discussion at the Congress would be impossible. This is particularly the case with the Feret work which

cannot be dealt with at the Congress with the required knowledge and profundity unless a previous study has been made of it.

The sub-commission also occupied itself with the question of a normal lime.

The German union for testing technological materials at its principal meeting in September 5th 1903 selected the lime of the united Harz lime industry at Elbingerode as normal lime. Prof. Gary expressed the wish that this lime should be proposed to the international union as normal lime.

This wish the undersigned carried out in a circular note to the members of our Commission. These gentlemen were also provided with a table showing the composition of those kinds of lime which had been submitted to Committee 6 of the German union for testing materials and there was also submitted to them the actual wording of the resolution in regard to the way that the normal lime is to be selected for Germany and the way in which it should be packed and transported.

I have taken the liberty of adding both these documents as Appendices *A* and *B*.

Seven answers have been received.

Prof. Gary says in his answer that he is convinced that in the Christina cliff we have a source for normal lime which cannot be surpassed anywhere. The same gentleman also informed me that the united Harz-works have built pits and slacking troughs according to his sketches. The Royal Mechanical Testing Institute in Gross-Lichterfelde has also taken up the subject and has directed a request to the minister in whose department the subject lies to empower them to take control of the normal lime in the same sense in which this has already been done in the case of normal sand. As soon as the decision of the minister has been received, which it is anticipated will be favourable, the slacking and taking of samples will be carried out under the supervision of Prof. Gary.

It is accordingly to be hoped that the first standard or normal lime will be put into commerce in the summer of 1904. A sample will be laid before the Brussels Congress so a to show the nature of the lime and the way it is packed.

Dr. Michaelis of Berlin states that he is in agreement with the proposals of my circular letter.

Herr Thiele, architectural councillor of Cassel, regards the determination of a normal lime by the researches of the international union for testing materials as a matter to be desired, and the lime selected by the German Institute as suitable.

Engineer Paul Wagner declares that he is also in agreement with the proposals of the circular letter.

Prof. van der Kloes agrees with the selection of the source, proposes, however, that the lime should be sent out not as dry hydrate of lime but as a paste.

In the dry condition it is stated (by him) that the lime is much easier to handle and can be slaked by people who understand their business in large quantities or in the Olchewesky slaking drum.

Mr. Feret expresses himself as opposed to the adoption of a standard lime. He specially points out that the employment of lime in the pasty condition is not so suitable for tests with puzzolanas as the use of lime in powder. It is more difficult to obtain a homogeneous mortar with lime in a pasty condition than when puzzolane is first mixed with powdered lime, sand added, and water then mixed with the materials when they have been first well blended dry.

In the case of a lime paste that has been preserved for a longer or shorter period in a case, one is never certain whether it possesses the same amount of water in all parts, even when the case is air-tight; this is particularly so when the case is not completely filled.

It would then be necessary in the case of every experiment to mix the lime paste most carefully and to determine the amount of water in order to know how much lime paste should be taken to make the required mortar and how much water should be added.

Mr. Feret is opposed on principle to the introduction of a monopoly in the supply of materials or apparatus for the normal experiments. The role of the international union consists only in determining with great accuracy the conditions which materials and apparatus must fulfil, without prescribing any particular brand or place of origin.

If the union deviate from this standpoint it can only lead to strife which would have the effect of delaying the realization of the end at which the union aims, viz., the attainment of unification.

In any given case it is easy to find a normal lime possessing the qualities required for the test with puzzolanas. The tests which he had made and submitted in his report to the Congress had shown that the composition of the lime might vary without producing noticeable variations in the qualities of the mortars made with it

Prof. Dr. Rebuffat is of opinion that it is not necessary to obtain the lime for experiments with puzzolana always from the same quarry and the same kiln. It is sufficient if the limes contain the same amount of CaO and foreign substances. Such limes are to be found in nearly all countries. In addition it would not be feasible in Italy to obtain the lime from such a distance on account of the expense. Besides, it was not shown by table B (Appendix) that the normal lime was the best of the limes tested.

To these expressions of opinion of the different members I would like to add that it has been proposed by Committee VI of the German Union to employ lime paste and not lime powder on account of the difficulty involved in slaking lime to lime-hydrate in such a way that a uniform powder is always produced without containing imperfectly slaked portions.

If, however, the lime is blended with water, it undergoes no alteration with perhaps the exception of a $1^1/_2$ mm thick layer at the top. In former years we have worked with lime paste only. In one quarry the whole year's output used to stand under water (till used). Its removal is easy, a shovelful is taken out and the top layer that has been softened by contact with the water is removed with a knife and the rest squeezed in a coarse cloth like that employed for wringing cheese in Switzerland.

This lime paste nearly always contains 52 to 55% of water. The water content is much more uniform than in the case of lime powder. In the case of the normal lime which is sent out in tins, after opening a little water can be poured on the paste. When

more lime is removed the water is first poured out and subsequently renewed.

The lime paste kept at the quarry has always given more regular results than lime powder even when the latter was kept in bottles with ground-in stoppers.

I hold it possible in large laboratories to prepare a uniform effective fat lime powder when the burnt lime used for the purpose is always drawn from the same source. But on works etc. while building is in progress and where the experiments are to be made, great difference in the manufacture of the lime powder will always occur and in spite of sieving lime will often get used that contains imperfectly slaked portions. The preservation of lime powder, too, is extremely difficult and the content of carbonic acid continues regularly to increase unless the most careful precautions are observed.

Among the few expressions of opinion on the normal lime question that have been received, affirmative replies came only from Germany. As there thus appears to be little chance of procuring the general acceptance of a normal lime, the question may at any rate be raised whether each country could not itself undertake to prepare its own normal lime. The differences in behaviour of these normal limes could be tested by the great testing institutes using the same additions. The acceptance of a lime paste as normal or standard lime would have the great advantage that:

1. Practically no errors can take place in slaking of the kind that so easily occur in the preparation of lime powder.

2. The preservation of lime under water keeps it quite uniform in its properties and prevents the formation of carbonate of lime with the exception of a thin layer at the top. (After lime powder has been kept for a length of time every experiment would have to be preceded by a determination of the content of carbonate of lime which is not only very tiresome and tedious but in building works etc. quite impracticable.)

The employment of lime paste also presents the great advantage that the experimenter is compelled to knead the mixture horoughly till it is no longer streaky. Just this intimate mixture of lime and puzzolana in making mortar is the very thing that is

never rightly understood or carried out by those who begin to experiment with puzzolana and have previously only been familiar with cement.

The favourable conditions prevailing in the case of the German normal lime will not be easy to find everywhere as they are not so much due to the plans of Prof. Gary for manufacture and packing as to the circumstance that these operations are supervised by the «Royal Testing Institute» (Königl. mech. techn. Versuchsanstalt) in Gross-Lichterfelde.

Chairman for problem 11

G. Herfeldt, Tufa-quarry owner.

P. S. Since, the Royal Office for Testing Materials (Königl. Material-Prüfungsamt) in Groß-Lichterfelde (formerly the „Royal Testing Institute) has taken over the control of manufacturing this lime. For sale, please apply to „Laboratorium für Tonindustrie" in Berlin, NW. 21, Dreysestraße 4.

Appendix A.

Limestones submitted for

The percentages in italics refer to the substance

No.	Sent in by:	Specif. gravity average of 2 tests	Weight per unit volume, in mass average of 4 tests	Degree of density	Condition of Fracture		
					Structure	Fracture	Colour
1	Rhein.-westfälische Kalkwerke, Dornap	2·715	2·699	0·994	Uniformly dense to crystalline	sharp-edged conchoidal	dark-grey
2	L. Kiepenheuer, Kalk- und Mörtelindustrie, Cologne. From the quarry at B.-Gladbach	2·709	2·691	0·993	Uniformly dense to crystalline	sharp-edged conchoidal	dark-grey
3	Vereinigte Harzer Kalkindustrie, Elbingerode (Harz)	2·713	2·696	0·994	Uniformly dense to crystalline	sharp-edged conchoidal	grey
4a	Ritter von Hertberg'sche Verwaltung d. Domäne Reichenau (Austria) (yellowish-white)	2·703	2·700	0·999	Uniformly dense	irregularly sharp-edged	yellowish-white
4b	ditto (bluish-grey)	2·715	2·701	0·995	Uniformly dense	sharp-edged conchoidal	brownish-grey
5	Neue Walheimer Kalkwerke A.-G., Walheim near Aix-la-Chapelle	2·703	2·699	0·999	dense to fine grain with veins of white calcareous spar	sharp-edged conchoidal	dark-grey
6	Zentr.-Verkaufsstelle der Setzdorfer Kalkwerke Setzdorf (Austr. Silesia)	2·703	2·691	0·996	Uniformly fine-grain, crystalline	irregularly sharp-edged	grey
7	Gebr. Leube, Ulm a. D., Kalk von Herrlingen, weißer Jura nach Quermstedt	2·703	2·646	0·979	Uniformly dense with calcareous spar inclusions	sharp-edged conchoidal	yellowish-white
8	Société anonyme des Carrières et Fours à Chaux de la Meuse in Liège (Belgium)	2·703	2·683	0·993	Uniformly dense	conchoidal sharp-edged	grey-black
9	Ancienne Firme Fallon Thierry in Namur (Belgium) from the pit at Saint-Nicolas	2·709	2·695	0·995	Uniformly dense	conchoidal sharp-edged	grey

making standard lime.

in a state free from water and from carbonic acid

Chemical composition						
Loss on ignition	Silica and insol. matter	Ferric oxide alumina etc.	Lime	Magnesia	Sulphuric anhydride	Undetermined (alkalis etc.)
43·99 %	0·46 %	0·19 %	54·49 %	1·07 %	0·08 %	—
—	*0·82 %*	*0·34 %*	*96·80 %*	*1·90 %*	*0·14 %*	—
43·47 %	1·42 %	0·18 %	53·67 %	0·85 %	0·11 %	0·30 %
—	*2·51 %*	*0·32 %*	*94·94 %*	*1·50 %*	*0·19 %*	*0·53 %*
43·89 %	0·40 %	0·24 %	54·87 %	0·18 %	0·07 %	0·35 %
—	*0·71 %*	*0·43 %*	*97·79 %*	*0·32 %*	*0·12 %*	*0·62 %*
44·01 %	0·11 %	0·15 %	55·48 %	0·34 %	0·03 %	—
—	*0·20 %* (Traces)	*0·27 %* (Traces)	*98·88 %* (99·27)	*0·61 %* (0·80)	*0·05 %*	—
43·99 %	0·18 %	0·20 %	55·18 %	0·50 %	0·07 %	—
—	*0·32 %*	*0·36 %*	*98·31 %*	*0·89 %*	*0·12 %*	—
44·18 %	1·75 %	0·20 %	53·91 %	0·28 %	0·04 %	—
—	*3·11 %*	*0·36 %*	*95·96 %*	*0·50 %*	*0·07 %*	—
43·90 %	0·15 %	0·12 %	55·60 %	0·28 %	—	—
—	*0·27 %*	*0·21 %*	*99·02 %*	*0·50 %*	—	—
43·71 %	0·22 %	0·34 %	55·49 %	0·28 %	0·06 %	—
—	*0·39 %* (0·23)	*0·60 %*	*98·40 %* (99·11)	*0·50 %* (0·63)	*0·11 %*	—
43·91 %	0·30 %	0·03 %	55·49 %	0·30 %	0·04 %	—
—	*0·53 %*	*0·05 %*	*98·80 %*	*0·53 %*	*0·07 %*	—
43·72 %	0·34 %	0·02 %	55·45 %	0·24 %	0·01 %	0·22 %
—	*0·60 %*	*0·04 %*	*98·53 %*	*0·43 %*	*0·02 %*	*0·40 %*

Annex B.

Conclusions regarding the future manner of delivery of standard lime.

It has been decided that the standard lime to be brought into commerce is to be in the form of lime-paste and not of pulverulent slaked lime. Before using this lime the moisture should be ascertained by evaporation, the amount of water in the mortar to be adjusted accordingly.

After inspection of the pit and the lime-burning factory «Christinenklippe», the conclusion to be submitted to the principal meeting is as follows:

The limestone from the quarry «Christinenklippe» at Rübeland belonging to the Vereinigte Harzer Kalkindustrie of Elbingerode is to serve as raw material for the production of standard lime.

Large pieces of this limestone, as pure as possible, are to be burnt in the ring kiln at Rübeland. From the burnt pieces, the purest are again selected and slaked as soon as all concretions, cinder etc. contained in them have been removed. When slaking, care should be taken that the water submerges the lime for some time, and that of the cream of lime the upper $^3/_4$ only are run into the pit in order to retain those parts which are slaked with more difficulty in the pan.

The cream of lime is run into a pit divided into two parts by a masonry partition, one of the chambers being always kept filled and closed, while from the other the standard lime to be sold is taken and enclosed in air-tight vessels of two sizes the dimensions of which are to be determined later.

The sale of the standard lime is undertaken by the chemical laboratory in connection with the Laboratorium für Tonindustrie, Berlin NW. Kruppstraße No. 6.

It is intended to establish an official station for controlling the manufacture of standard lime on the same lines as adopted for Prussian normal sand.

The determination of limits of error is not being considered at present, until experience has shown with what constancy normal or standard lime, can be produced on a large scale.

Tests are to be undertaken to show whether fresh or old lime-pastes, stored under water, show different qualities as regards strength and tensile strain.

Annex C.

Methods of Testing Puzzuolana.

Preliminary tests by R. Feret—Boulogne-sur-Mer. Carried out by **Gabriel Herfeldt**, Andernach on the Rhine.

A. Tests for ascertaining

the influence of the fineness on the setting and the hardening of puzzuolana (trass) mortar.

In order to be, as far as possible, in accordance with practical conditions, trass of the following fineness was used in the tests:

Series I. Trass leaving 30% residue on a 900 mesh sieve
» II. » » 20% » » » 900 » »
» III. » » 0% » » » 900 » » *)
» IV. Trass the residue of which left on 5000 or 900 mesh sieves respectively, was subsequently crushed to such an extent as to allow the whole to pass a 5000 mesh sieve.

B. Selection of the lime.

For these tests the so-called Liege fat lime from the limestone quarries at Moha near Namur, noted for its purity, was used.

(98·53% pure *CaO*.)

It was used in the form of powder.

The slaking of the lime and the storing of same before use took place in accordance with the recognised methods of the German association for testing materials.

*) This grade of fineness was obtained by using the 900 mesh sieve. The entire residue was subsequently crushed over and over again until the whole passed this sieve. 66% of this trass passed the sieve with 5000 meshes.

Slaked lime-powder *a*

Only such lime was used as gave no residue on the 900 mesh sieve: the remaining portions were sifted off and thrown away.

Slaked lime powder *b*.

Only the residue on the sieve with 120 mesh sieve was removed.

C. Composition of the sand used for the test.

For these tests sand from the Rhine was used, having the degree of fineness proposed by R. Feret, i. e. equal parts in weight of the following three sizes of grain:

1. Grains—passing through a sieve with holes of 2 mm diam.
   but remaining on » » » $1^1/_2$ » »
2. Grains—passing through a sieve with holes of $1^1/_2$ mm diam.
   but remaining on » » » 1 » »
3. Grains—passing through a sieve with holes of 1 mm diam.
   but remaining on » » » $^1/_2$ » »

D. The following proportions of puzzuolana (trass), lime, and sand were used:

1. For the setting tests: (needle tests) by means of the standard needle-apparatus, as used by Professor L. v. Tetmajer. Observations were taken as to the gradual hardening with a needle 1 mm in diameter and loaded with 300 g.

Mixture of parts by weight.

a) 2 parts of puzzuolana (trass)
1 part of lime-powder (80 g; 40 g) and

b) 3 parts of puzzuolana (trass)
1 part of lime-powder (120 g; 40 g)

with the trasses of different fineness.

2. For the tests of strength.

Mixture of parts by weight:

a)	2 parts of puzzuolana (trass)	1000 g
	1 part of lime-powder	500 »
	3 parts of sand of the prescribed consistency .	1500 »
and *b*)	2 parts of puzzuolana (trass).	1000 »
	$1^1/_2$ » of lime-powder	750 »
	6 » of sand — as above —	3000 »

From *a* and *b* of the normal consistency.

For the test with a larger proportion of water, for the tests for strength, only mixture *a* with slaked lime-powder *a*.

E. The mixing of the mortars

was as thorough as possible until no streaks due to unmixed lime were visible. The puzzuolana (trass) and the lime-powder were mixed in the dry state until a uniform colour was obtained, before adding sand and water.

F. Quantity of water for the standard (or normal) mortar having regard to a constant consistency.

Here the proposals of Mr. R. Feret were exactly followed. The little rod of the consistency-tester sinks by its own weight into the box filled with mortar till it comes to a standstill. 6 mm above the bottom surface of the box; thus the layer of mortar not pierced must have a thickness of 6 mm.

G. With the individual tests of mortar of greater plasticity (larger proportion of water), in all cases 10% more*) water was added than was found in standard (or normal) mortar.

H. For the tests the following kinds and the following number of samples were prepared.

1. Setting tests (needle tests) 1 sample each.

Hardening took place under fresh water immediately after manufacture.

The samples were examined for progressive hardening after 2, 3, 4 and 5 days.

2. Tests for strength. 10 samples of each mixture prepared for the tensile test, 2 samples of each mixture prepared for the compression test.

Manner of hardening: 1 day in moist air, i. e. air saturated with water in a well-closed zinc box (on the average 92% saturation), then under fresh water.

*) A still larger proportion of water was impossible having regard to the successful preparation of the test pieces and to their removal from the moulds.

Testing times: after total hardening of 1 + 13 days, and after a total hardening of 1 + 27 days.

Manner of preparation: All samples were subjected to the impact of a Böhme's hammer 150 times.

The tension test samples were removed from the moulds soon after their preparation; but in the case of the samples for the pressure test, the moulds were not removed till after 24 hours in order to preserve the surfaces exact.

I. The temperature of the water

was on the average, in the case of setting tests (needle tests) during the period of storage of 5 days = 15° C. In the case of the tests of strength (tensile and compression tests) for the period of 14 days = $17^3/_4{}^0$ C., for the period of storage of 28 days = 18° C.

K. Testing materials.

For puzzuolana, that from the Rhine was obtained from the Nette valley, from Gerhard Herfeldt's trass or tufa quarries at Plaidt near Andernach.

For lime, the above mentioned Liège fat lime from Moha near Namur was used, in form of powder.

For sand, sand from the Rhine was used of the composition proposed by R. Feret.

L. In the case of the setting tests

(needle tests) — as already remarked — the standard needle-apparatus introduced by Professor L. v. Tetmajer was used.

In the case of the tests of strength

a) Tensile tests — Dr. Michaelis' standard testing-apparatus.

b) Compression tests — the Amsler-Laffon 30 ton press respectively were used.

Carrying out the tests.

I. Setting tests. — (Needle tests.)

1. With slaked lime powder *a* ($0^0/_0$ residue on the 900 mesh sieve)

and the trasses: I, II, III, IV.

Marks of samples: | || ||| ||||

Mixture: 2 parts in weight of puzzuolana (trass) = 80 gr.
1 „ „ „ „ lime-powder . . = 40 „
+1 „ „ „ „ water . . . . = 40 „

dto. like I, II, III, IV.

Marks of samples / // /// ////

and 3 parts in weight of puzzuolana (trass) = 120 gr.
1 „ „ „ „ lime-powder . . = 40 „
$+1^1/_3$ „ „ „ „ water . . . . = 53 „

2. with slaked lime-powder *b* (only removed the residue remained on the 120 mesh sieve)

and the trasses

and in the mixtures as before.

Marks of samples 1, 2, 3, 4, 5, 6, 7 and 8.

With lime powder a.

Trass respectively marks of samples	I		II		III		IIII		/		//		///		////	
Test age	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration
2 days:	300	1/2	300	1/2	300	1/2	300	1	300	1/2	300	1	300	1	300	1/2
	800	3 1/2	800	2 1/2	800	2	800	3	500	1 1/2	500	1 1/2	500	1 1/2	500	1 1/2
	1000	5	1000	5	1000	3	1000	4 1/2	800	2 1/2	800	3	800	3	800	2
	1300	7	1300	7	1300	4 1/2	1500	5	1000	4 1/2	1000	4 1/2	1000	4	1000	3
	1500	8 1/2	1500	8	1400	5	1800	7	1050	5	1100	5	1300	5	1300	4
					1800	8			1300	6	1300	6 1/2	1500	7	1500	4 1/2
									1500	8 1/2	1500	9			1800	5
															1900	5 1/2
3 days:	1300	1 1/2	1300	1 1/2	1300	1	1300	1 1/2	1300	2	1300	2	1300	2	1300	1 1/2
	2000	4	2000	3 1/2	2000	3	2000	3 1/2	1500	3	1500	3	1500	3	1500	2 1/2
	2300	5	2300	4 1/2	2300	3 1/2	2300	4	2000	4 1/2	2000	4	2000	4	2000	3 1/2
	2500	6	2400	5	2500	4	2500	5	2200	5	2300	5	2300	4 1/2	2500	4
			2500	6	2800	5	2700	6	2500	9	2500	8	2500	5	2800	5
					3000	6							2600	6	3000	6
4 days:	2500	2	2500	2 1/2	2500	2 1/2	2500	3	2500	2 1/2	2500	3	2500	3	2500	2
	3000	3 1/2	3000	3 1/2	3000	3 1/2	3000	4	3000	3 1/2	3000	4	3000	4	3000	3
	3500	5	3500	5	3500	5	3400	5	3500	5	3300	5	3500	5	3500	5
	3800	5 1/2	3800	6 1/2	3800	6 1/2	3500	5 1/2	3800	5 1/2	3500	5 1/2	3800	6	3800	5 1/2
							3800	7			3800	7				
5 days:	3000	2	3000	2	3000	1 1/2	3000	1/2	2500	1 1/2	2500	2	2500	2	2500	1 1/2
	4000	3 1/2	4000	3	4000	3	4000	3 1/2	3000	2 1/2	3000	3	3000	3	3000	2 1/2
	5000	5	5200	5	5000	4	6000	5	3500	3 1/2	3500	3 1/2	3500	3 1/2	3500	3
	5300	6	5500	6	5500	5	6500	8	4000	4 1/2	4000	4	4000	4	4000	3 1/2
					5800	6			4200	5	4500	5	4500	4 1/2	4500	4 1/2
									4500	6	4600	6 1/2	4600	5	4600	4 1/2
													4600	5 1/2	4700	5
															4800	6

With slaked lime powder b.

Trass respectively marks of samples	1		2		3		4		5		6		7		8	
Test age	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration	Load	Penetration
2 days:	300	1/2	300	1/2	300	1 1/2	300	1	300	1/2	300	1/2	300	1/2	300	1/2
	500	2	800	2	500	2	500	2 1/2	500	1	500	1 1/2	500	1	500	1
	800	3 1/2	1000	3	1000	3 1/2	1000	3	1000	4	1000	4	1000	3	1000	3
	1000	4	1300	5	1300	4 1/2	1300	4	1100	5	1100	5	1300	5	1300	5
	1200	5	1500	7	1500	5	1500	4 1/2	1300	6	1300	6	1500	6 1/2	1500	6 1/2
	1300	6			1800	7	1600	5	1500	8	1500	8				
	1500	8 1/2					1800	6								
3 days:	1300	3	1300	2 1/2	1300	1 1/2	1300	1 1/2	1300	3	1300	3 1/2	1300	2	1300	2
	1500	4	1500	3	1500	2	1500	2	1500	4	1500	4	1800	3	1800	2 1/2
	1800	5	1800	4	2000	3 1/2	2000	3	1800	5	1800	5	2000	3 1/2	2000	3
	2000	6	2000	5	2300	4 1/2	2500	4	2000	6	2000	6	2300	5	2500	5
			2300	5 1/2	2500	5	2800	5					2500	6 1/2	2800	6 1/2
			2500	6	3000	8	3000	6 1/2								
4 days:	2000	3	2000	2	2500	2 1/2	2500	2 1/2	2500	3	2500	3	2500	2	2500	2
	2300	4	2500	3	3000	3	3000	3 1/2	3000	4	3000	4	3000	3	3000	3
	2500	5	2800	3 1/2	3500	4	3500	4 1/2	3500	5	3500	5	3500	4	3500	4
	2800	6	3000	5	4000	5	4000	5	4000	6 1/2	4000	7	3800	5	3800	5
			3300	6 1/2	4500	6	4500	6					4000	5 1/2	4000	5 1/2
5 days:	2500	2	2500	1 1/2	2500	2	2500	2	2500	2	2500	2	3000	2	3000	1 1/2
	3000	2 1/2	3000	3	3000	3	3000	3	3000	3 1/2	3000	3	3500	2 1/2	3500	2
	3500	4 1/2	3500	3 1/2	3500	4	3500	3 1/2	3500	4	3500	3 1/2	4000	3 1/2	4000	3 1/2
	4000	5	4000	5	4000	4 1/2	4000	4	4000	4 1/2	4000	4	4500	4	4500	4
	4500	6	4500	5 1/2	4500	5	4500	5	4500	5	4500	5	5000	5	5000	5
					5000	5 1/2	5000	5 1/2	5000	6 1/2	5000	5 1/2	5300	5 1/2	5300	5 1/2

II. Tests of strength (Tensile and compressive Tests).

A. With normal consistency.

1. With slaked lime-powder *a* (0% residue on the 900 mesh sieve).

Mixture: 2 parts in weight of puzzuolana (trass) . . . 1000 g
1 part in weight of lime-powder . . . . . 500 »
3 parts in weight of sand from the Rhine, composition as prescribed . . . . . 1500 »
+ $1^1/_{16}$ (circ.) parts in weight of water . . 530 »
and the trasses I, II, III and IV.

Marks of samples 1, 2, 3 and 4.

These were prepared for each 10 tensile test samples, 5 after 14 days, 5 after 28 days, and 2 compressive test samples, 1 after 14 days, 1 after 28 days.

As before.

Mixture: 2 parts in weight of puzzuolana (trass) . . 1000 g
$1^1/_2$ parts in weight of lime-powder . . . . 750 »
6 parts in weight of Rhine sand, combined as prescribed . . . . . 3000 »
+ (circ.) $1^1/_4$ parts in weight of water . . 620 »
and the trasses I, II, III and IV.

Marks of samples 5, 6, 7 and 8.

There were prepared 10 tensile test samples, 5 after 14 days, 5 after 28 days, 2 compressive test samples, 1 after 14 days, 1 after 28 days.

Results of the strength tests.

Test samples	Mark 1	Mark 2	Mark 3	Mark 4	Mark 5	Mark 6	Mark 7	Mark 8
Tensile strength kg/cm²	kg 13·54	kg 14·81	kg 15·30	kg 16·04	kg 13·88	kg 14·32	kg 16·20	kg 17·18
Average from 5 test samples Age: 1 + 13 days:	weight 652 gr.	weight 650 gr.	weight 642 gr.	weight 630 gr.	weight 683 gr.	weight 680 gr.	weight 672 gr.	weight 662 gr.
Compression test strength kg/cm²	kg 52	kg 52	kg 54	kg 56	kg 52	kg 56	kg 58	kg 67
Age: 1 + 13 days:	weight 685 gr.	weight 685 gr.	weight 675 gr.	weight 660 gr.	weight 715 gr.	weight 715 gr.	weight 705 gr.	weight 687 gr.
Tensile strength kg/cm²	kg 17·63	kg 17·84	kg 18·50	kg 19·36	kg 16·75	kg 18·00	kg 19·31	kg 20·84
Average from 5 test samples Age: 1 + 27 days:	weight 655 gr.	weight 655 gr.	weight 645 gr.	weight 635 gr.	weight 685 gr.	weight 680 gr.	weight 675 gr.	weight 662 gr.
Compression test strength kg/cm²	kg 63	kg 67	kg 77	kg 77	kg 73	kg 73	kg 79	kg 78
Age: 1 + 27 days:	weight 685 gr.	weight 685 gr.	weight 675 gr.	weight 665 gr.	weight 715 gr.	weight 715 gr.	weight 705 gr.	weight 705 gr.

2. With slaked lime-powder *b* (removing only the remainder on the 120 mesh sieve).

Mixture:	2 parts in weight of puzzuolana (trass) . .	1000 g
	1 part in weight of lime-powder	500 »
	3 parts in weight of Rhine sand, combined as stated	1500 »
	+ $1^1/_{16}$ (circ.) parts in weight of water . .	0·530 »
	and the trasses series I, II, III and IV.	
	Marks of samples 9, 10, 11 and 12.	

There were prepared 10 tensile test samples, 5 after 14 days, 5 after 28 days, 2 compressive test samples, 1 after 14 days, 1 after 28 days.

As before.

Mixture:	2 parts in weight of puzzuolana (trass) . .	1000 g
	$1^1/_2$ parts in weight of lime-powder	750 »
	6 parts in weight of sand, combined as described	3000 »
	+ circa $1^1/_4$ parts in weight of water . .	0·620 »
	and the trasses series I, II, III and IV.	
	Marks of samples 13, 14, 15 and 16.	

There were prepared 10 tensile test samples, 5 after 14 days, 5 after 28 days, 2 compressive test samples, 1 after 14 days, 1 after 28 days.

Results of the strength tests.

Test samples	Mark 9	Mark 10	Mark 11	Mark 12	Mark 13	Mark 14	Mark 15	Mark 16
Tensile strength kg/cm²	kg 16·80	kg 17·25	kg 18·47	kg 19·87	kg 15·92	kg 16·20	kg 16·49	kg 17·78
Average from 5 test samples								
Age: 1 + 13 days:	weight 660 gr.	weight 650 gr.	weight 645 gr.	weight 630 gr.	weight 675 gr.	weight 675 gr.	weight 672 gr.	weight 660 gr.
Compressive strength kg/cm²	kg 60	kg 63	kg 64	kg 65	kg 70	kg 71	kg 70	kg 73
Age: 1 + 13 days:	weight 680 gr.	weight 680 gr.	weight 680 gr.	weight 670 gr.	weight 715 gr.	weight 715 gr.	weight 710 gr.	weight 705 gr.
Tensile strength kg/cm²	kg 20·36	kg 20·06	kg 21·98	kg 22·84	kg 19·76	kg 20·18	kg 20·03	kg 21·75
Average from 5 test samples								
Age: 1 + 27 days:	weight 660 gr.	weight 650 gr.	weight 650 gr.	weight 635 gr.	weight 675 gr.	weight 680 gr.	weight 672 gr.	weight 665 gr.
Compressive strength kg/cm²	kg 82	kg 82	kg 83	kg 82	kg 81	kg 79	kg 85	kg 78
Age: 1 + 27 days:	weight 685 gr.	weight 680 gr.	weight 680 gr.	weight 670 gr.	weight 715 gr.	weight 715 gr.	weight 710 gr.	weight 705 gr.

B. With larger proportion of water.

(10% more water than at the normal consistency)

Mixture: 2 parts in weight of puzzuolanas (trass) . 1000 gr.
1 part in weight of limepowder *a* . . . 500 „
3 parts in weight of sand from the Rhine in the described composition . . . . . 1500 „
+ about $1^1/_6$ parts in weight of water . . 585 „
and the trass sorts: I., II., III. and IV.
marks of samples: 17, 18, 19 and 20.

There were prepared:
10 tensile samples, 5 after 14 days, 5 after 28 days
2 compressive test samples, 1 „ 14 „ 1 „ 28 „

Results of the strength tests:

Test samples	mark 17	mark 18	mark 19	mark 20
Tensile strength kg/cm² Average from 5 test samples. Age: 1 + 13 days:	kg 15·42 weight: 650 gr.	kg 17·10 weight: 660 gr.	kg 19·32 weight: 645 gr.	kg 19·21 weight: 635 gr.
Compressive strength kg/cm² Age: 1 + 13 days:	kg 55 weight: 675 gr.	kg 54 weight: 680 gr.	kg 74 weight: 675 gr.	kg 72 weight: 670 gr.
Tensile strength kg/cm² Average from 5 test samples. Age: 1 + 27 days:	kg 19·10 weight: 650 gr.	kg 20·76 weight: 665 gr.	kg 20·12 weight: 645 gr.	kg 21·95 weight: 635 gr.
Compressive strength kg/cm² Age: 1 + 27 days:	kg 72 weight: 675 gr.	kg 77 weight: 680 gr.	kg 85 weight: 675 gr.	kg 97 weight: 675 gr.

Some Concluding Tests

corresponding exactly with the proposals made by Mr. R. Feret.

There were used:

Plaidt standard trass
(leaving 50% residue on a 5000 mesh sieve);
so-called Liege slaked fat lime-powder *a*
from the limestone quarries at Moha near Namur
(leaving 0% residue on a 900 mesh sieve),
containing: 98·53% pure CaO;
Proportion of water: 24·22%.
Sand from the Rhine: The same composition as described in „Methods of Testing puzzuolana C".

I. Setting tests (needle tests).

Mixture of parts by weight:

200 gr slaked lime-powder *a*
1 part . . . . . ($^1/_5$ of it) 40 gr
800 gr Plaidt standard trass
4 parts . . . . . 160 „
+ 275 gr water . . . . . 55 „

Tests after 2 days		Tests after 3 days		Tests after 4 days		Tests after 5 days		Température of the water in hardening
Load in gr	Deflection in mm	Load in gr	Deflection in mm	Load in gr	Deflection in mm	Load in gr	Deflection in mm	
								2 days = 18° C.
300	2	500	1	1500	3	1900	$2^1/_2$	3 days = 19° C.
400	$2^1/_2$	1000	3	1700	4	2400	$4^1/_2$	
500	4	1300	4	1800	$4^1/_2$	*2500*	*5*	4 days = 18½° C.
600	*5*	*1500*	*5*	*1900*	*5*	2600	6	
700	6	1600	$6^1/_2$	2000	7			5 days = 18° C.

II. Strength tests (Tension and pressure tests).

Mixture:	50 g of slaked lime-powder *a*	200 g
	200 » of Plaidt standard trass	800 »
	750 « of Rhine sand, combined as described .	3000 »
	+ 103 » of water	412 »

There were prepared 10 tensile test samples, 5 after 14 days, 5 after 28 days, 2 pressure test samples, 1 after 14 days, 1 after 28 days.

Manner of hardening: 7 days in wet air (92% saturation) 7 and 21 days respectively under distilled water.

Manner of preparation: The mortar was prepared of the normal, consistency (trass and lime-powder) mixed dry previously as shown under F of the general tests, and forced into the moulds by 150 blows with Böhme's hammer-apparatus.

In the case of the tensile test samples the moulds were removed at once after their preparation, in the case of the pressure test samples after 24 hours.

Results of the strength tests.

Tensile strength Average from 5 samples Age: 7 + 7 days:	kg/cm² 9·13 kg weight: 695 g	Tensile strength Average from 5 samples Age: 7 + 21 days:	kg/cm² 10·10 kg weight: 695 g	Average Temperature of water during the 14 days trials	18¹/₄° C.
Compressive strength Age: 7 + 7 days	kg/cm² 50 kg weight: 710 g	Compressive strength Age: 7 + 21 days	kg/cm² 73 kg weight: 710 g	Average temperature of water during the 28 days trials	18¹/₄° C.
				Relative degree of moisture of air during the 7 days storage	92%

General Conclusions from the Results of the Tests.

1. Setting tests (needle tests).

Slaked lime-powder *a*.

While the tests with finer puzzuolana (trass) at the beginning of its hardening (after 2 and 3 days) mixture of parts in weight,

2 tr., 1 l., 1 w., as also

3 » 1 » $1^1/_3$ »

give higher degrees of hardness (resistance against increasing load), the results of the same after 4 days hardening, in the case of both mixtures, are nearly the same.

The test after 5 days gives under the same conditions higher values for the mixture with less trass (2 : 1 : 1) essentially increased strength as compared with the mixture with more trass (3 : 1 : 1).

In conclusion it may be assumed that the finer the trass the greater its binding power. The influence of the fatter mortar, compared with the more meagre one, is only shown after 5 days' hardening, with increasing load.

Slaked lime-powder *b*.

The tests with this confirm the supposition as with lime-powder *a*, that greater fineness results in higher values for the hardening in respect of the resistance against load.

The figures found are in general lower than those with lime-powder *a*.

This statement justifies therefore the conclusion, as with trass, that the finer the material is ground, the greater its setting power, because the finer lime-powder *a* (with 0% residue on the 900 mesh sieve) showed in all cases greater values throughout than with the coarser powder *b* (with 0% residue on the 120 mesh sieve).

In this case, contrary to the results with lime-powder *a*, after 5 days hardening the resistance to load with the poorer mortar 3 : 1 : 1 is greater than with the fatter one (2 : 1 : 1).

2. Tests of strength (Tensile and compressive tests).

A. With normal consistency.

Slaked lime-powder *a*.

When testing the strength the increasing fineness of the ground trass has a favourable influence on the hardening power of the puzzuolane (trass) mortar. The figures obtained may be considered as very favourable. Those samples, with the larger percentage of sand (having parts by weight: 2 tr., $1^1/_2$ l., 6 s. or by volume $1 : 1^1/_2 : 2$), showed almost greater strength than with the fatter mixture (2 tr., 1 l., 3 s. or by volume 1 : 1 : 1).

The results of the tension and pressure tests agree with the results of the experiments previously made by various experimenters, in so far as they relate to the samples containing 2 or at the most $2^1/_2$ parts by volume of sand, with especial reference to samples which have undergone the more extended period of hardening (one month and over). These results show that those samples with the larger proportion of sand (within the limits stated) give higher values than those containing only 1 or $1^1/_2$ parts by volume of sand.

Although the larger the proportion of sand the less the closeness of puzzuolana (trass) mortars, still the greater the quantities of sand, especially of sharp-grained sands present, the larger is the number of adhesion surfaces provided — with the result that in the tensile tests this shows itself as increased adhesion-moment of resistance, and in the case of the pressure tests as increased compression — moment of resistance — this in the latter case appearing as increased inherent strength.

Slaked lime-powder *b*

The results of this series of tests were still more favourable than with slaked lime-powder *a*. It seems accordingly correct to assume that with the coarser lime meal ($0^0/_0$ residue on the 120 mesh sieve) the moment of adhesion and the compression-strength has increased as in the case of the sample containing lime-

powder *a* and the larger proportion of sand, and that thus the tractive and compression strengths have been influenced advantageously (in contrast to the experience obtained with setting tests).

3. Tests with larger proportion of water (with slaked lime-powder a).

The figures of this series of tests — without regard to slight irregularities — increase directly as the puzzuolana (trass) becomes finer. Although the results may be yet somewhat better than those of the normal consistency, the preparation of the samples was very difficult, due to the high adhesive properties of the puzzuolane (trass) mortar and its great softness, and many samples (especially the samples for the pressure test) had to be repeatedly prepared in order to get complete series.

Concluding Tests — corresponding exactly with the Proposals made by R. Feret.

With normal consistency.

The proportions used were:

a. For the setting test:

1 lime-powder, 4 puzzuolane (trass) by weight or $^2/_5 : ^4/_5$ by volume and further:

b. for the tension and pressure tests:

1 lime-powder, 4 puzzuolane (trass), 15 sand by weight or $^2/_5 : ^4/_5 : 2$ by volume.

It must, however, be taken into account that the tests were executed at a higher temperature (18^0 C.); for lower temperatures such poor mixtures would not be advisable for testing the trass as the latter hardens only very slowly with lime at low temperature.

General Remarks and Proposals resulting from the Tests made.

As regards the question of uniform methods of testing puzzuolane*), the results obtained with the Rheinish puzzuolana (trass) from the Nette Valley near Andernach on Rhine have proved

*) Puzzuolana and trass are practically the same species of volcanic rock as is more generally known in Great Britain as tufa, trass being in particular the felsitic tufa of the Rhine and Laacher See. (Translator).

that the increase of the fineness influences perceptibly the hydraulic properties of Rhineland trass, and that increasing fineness gives increasing strength.

But practically the finer grinding of really genuin commercial trass, containing about 30% residue on the 900 mesh sieve, is very difficult, if as in the present case the residue is to be crushed in the mortar in small quantities until we get 20% and 0% on the residue respectively with 900 and 5000 mesh sieves.

I propose therefore to accept as the standard of fineness that material which gives 30% or at best only 20% residue on the 900 mesh sieve.

As regards the question of standard lime some special explanations and arrangements require to be made.

As far as the negotiations to this end have proceeded, it seems to be most advisable for each country, after having fixed precise rules for standard lime, to select the standard for itself.

As regards the influence of the fineness of the lime dust or powdered lime on the hardening of puzzuolana (trass) mortar, see the conclusions mentioned above. The composition of the sand, as proposed by R. Feret, appears to me very advantageous and very nearly in accord with practice; the preparation, too, of such sand in larger quantities is easily made with corresponding sieves of larger sizes.

As regards the proportions of mixtures for testing Rheinish puzzuolana (trass) I, consider that the most suitable are: for the setting tests (needle tests) when using fat lime in form of powder those without sand having a composition of (taking parts by weight): 2 of trass, 1 of lime-powder and 1 of water, and for tension and pressure tests a composition of: 2 parts of trass, 1 part of lime-powder, 3 parts of sand, 0·9 to 1 part of water by weight. When using fat lime as paste, in corresponding proportions for setting tests and strength tests, the use of larger proportions of lime or sand seems to be indicated as a line on which collateral tests may with advantage be carried out.

Andernach, Dec. 31st 1903.

(Signed) **Gabriel Herfeldt**
Chairman of the Committee for Problem 11.

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT OF THE COMMISSION FOR THE
STUDY OF PROBLEM 11.

Group B

TO ESTABLISH METHODS FOR TESTING PUZZUOLANAS WITH THE OBJECT OF DETERMINING THEIR VALUE FOR MAKING MORTARS.

ANNEX.
EXPERIMENTS MADE IN THE „LABORATOIRE DES PONTS ET CHAUSSÉES DE BOULOGNE-SUR-MER“ WITH THE VIEW OF DETERMINING THE METHODS FOR TESTING PUZZUOLANAS.

REPORT BY R. FERET.

(TRANSLATED FROM THE FRENCH ORIGINAL.)

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Experiments made in the „Laboratoire des Ponts et Chaussées de Boulogne-sur-Mer"

with the view of determining
methods for testing puzzuolanas.

Report by R. Feret.

Translated from the French Original.

Commission No. 11 sitting at Budapest on September 11th, 1901, in pursuance of its duty of fixing uniform rules for testing puzzuolana, adopted the following two principles:

1. The trials of strength must be executed with mortars mixed to a plastic state.

2. Besides the tests intended to supply information on the intrinsic qualities of puzzuolanas, for which tests the puzzuolanas must be reduced to a constant and extreme degree of fineness, other tests should be arranged in which the puzzuolanas are left in the somewhat coarse state in which they are used in construction.

The writer of this article was charged by the commission to prepare a programme of experiments with a view to discover the best way of applying the above conditions. Consequently, M. G. Herfeldt, the President of the Commission, received from him at the beginning of 1902, a series of proposals which after various preliminary experiments appeared to him to define the best methods of testing puzzuolana. This programme of testing puzzuolana,

printed in both French and German, was brought to the notice of the International Commission by its organ[1]). Separately printed copies were sent to the members of Commission No. 11.

The writer began by dividing the tests into two categories.

1. The characteristic tests, suitable for ascertaining the intrinsic properties of a puzzuolana, and for the comparison of different puzzuolanas with reference to those properties. These tests must be carried out under well defined conditions, identical for all samples. These are the standard tests proper.

2. Practical tests, specially designed to inform every builder of the results which he can obtain in practice with the puzzuolanas he uses. Some latitude must be allowed in these tests, taking into consideration the special circumstances prevailing in each building yard.

The programme has provisionally fixed for the preliminary experiments the conditions briefly stated below. These should be adhered to as closely as possible, except in the case of the special factor, the influence of which is the subject of investigation.

1. The Characteristic Tests.

A, Powder the puzzuolanas till 25 to 50 per cent will remain on a sieve of 4900 meshes to the cm^2

B, Use a powdered well-slaked fat lime, containing not more than 2 per cent of solid impurities, between 24·3 and 28 per cent of water (reckoned on the pure lime), and which has passed through a sieve with 900 meshes to the cm^2

C, Use a standard silicious sand with rounded grains, composed of equal weights of sizes of grain limited respectively by round holes of 2 mm, 1·5 mm, 1 mm and 0·5 mm, in diameter.

D, For tests on pastes without sand, take powdered fat slaked lime mixed with four times its weight of puzzuolana.

For tests on sand mortars, mix the above mixture with three times its weight of sand. We shall then have the proportion: Lime : puzzuolana : sand = 1 : 4 : 15 = 50 : 200 : 750.

[1]) Les matériaux de construction, 7th year. Numbers 1—2, 3—4, 7, 13, 15. Baumaterialienkunde, 7th year. Numbers 8, 10, 11, 12, 13, 15.

E, Mix the samples of mortar to a plastic consistency, taking each time one kg of dry mixture and working it up with the trowel on a non-absorbent surface. Fill the moulds by means of trowel and finger.

F, Determine the proportion of water for mixtures without sand by means of the consistency tester adopted by Sub-Commission B of Commission 22.

G, Deduce by a formula from this proportion of water the proportion of water to be used in mixtures containing sand.

H, Make the setting test with the standard paste by means of the Vicat needle, according to the method adopted by Sub-Commission B of Commission 22.

I, Make the strength tests of the standard sand-mortar by compressing either cubes or prisms with bases 4 cm^2 previously broken by bending.

K, Keep the samples of mortar seven days in air saturated with moisture, and then 3 weeks in soft water.

2. Practical Tests.

L, Determine the percentage of the grains which will not pass through holes of 20, 10, 5, 2 and 0·5 mm in diameter nor through sieves with 900 and 4900 meshes per cm^2.

M, Tests of homogeneity, in which the puzzuolanas are divided, according to their coarseness, into classes which are examined separately.

N, Tests of strength with various mixtures of lime, puzzuolana and sand, which materials may be the same as those actually used in the yard.

O, Setting tests, by means of the thumb, with sand mortars.

P, Tests of deformation on mortars in various media.

Q, Tests of adhesion, by bending of prisms formed of two parts, one being an adhesion-body upon which the other, a mortar, is moulded.

To the programme was annexed a report of preliminary experiments justifying the proposals contained in it. To these we shall not refer in the present report.

We shall now describe the new experiments in conformity with this programme made in the Bridge and Highway Laboratory at Boulogne.

Except in certain cases, which will be pointed out, the above programme was strictly followed. The fat limes used were, however, generally less pure than defined in B.

Parallel tests were often made, also, by the methods adopted by the German association for testing technical materials (Deutscher Verband für die Material-Prüfungen der Technik).

The tests are grouped under the headings of letters, according to the object of each series.

I. The Characteristic Tests.

A. Effect of the degree of coarseness of the puzzuolana.

1st Test. Some slightly baked gaize*) was broken up and passed through holes 3mm in diameter. The residue was crushed again until it passed entirely through these holes, and the whole was mixed homogeneously. The powder was then weighed out in equal lots and ground in a ball-mill making the following numbers of revolutions:

Sample	A	B	C	D	E	F
Revolutions	200	400	600	1000	2000	10.000

Several other similar lots, each ground in 400 revolutions and of which 34 per cent would then remain on a sieve with 4900 meshes to the cm^2, were mixed together and passed through this sieve.

A portion of the fine powder thus obtained was reground in 8000 revolutions and 66 parts of the new powder were mixed with 34 parts of the residue. In this way a sample marked H, was obtained differing only from sample B by the finer size of the powder which had been through the 4900 sieve.

Another portion of the residue was reground, and then freed from all the grains retained by the 900 sieve, and from those passed through the 4900 sieve. By mixing 34 parts of these grains (900—4900) with 66 parts of the fine initial powder, a sample marked K was obtained, only differing from B by the fineness of the residue from the 4900 sieve.

Finally a sample marked L was prepared by crushing the original powder by impact and removing as it was produced everything that passed the 4900 sieve. When the pulverisation was judged sufficient, these dusts were mixed with the rest. The result

*) Gaize is a very friable argillaceous sandstone found in the Ardennes and the Meuse valley. (Note of the Translator).

was a powder of 22 per cent residue on the 4900 sieve, and was nearly the same as sample C, and only differing from it in the fact that its fine powder had undergone a less prolonged pulverisation.

These nine samples were tested in the ordinary way by sifting, and also by levigation, in ascending vertical currents of water having velocities of 2, 10 and 30 cm per minute respectively. The proportions of the residues left were as follow:

Designation of the gaize:			A	B	C	D	E	F	H	K	L
Sifting Tests	Total proportion remaining on a sieve of	4900 meshes	55	34	22,5	10,5	8	3,5	34	34	22
		900 meshes	2	6	2,5	1,5	1	2	6	0	7
		324 meshes	17,5	4	2	0,5	0,5	0,5	4	0	6
		144 meshes	11	2,5	0,5	0,3	0,2	0	2,5	0	1
Levigation Tests	Total proportions of grains not carried away by a current of	2 cm per min.	79,3	71,9	64,8	49,1	46,4	28,8	46,2	72,4	66,9
		10 cm per min.	46,9	52,5	39,7	26,4	14,0	8,0	38,2	49,1	41,3
		30 cm per min.	45,6	17,8	1,7	0,8	0,5	0,2	22,9	14,1	7,6

Mixtures were made of a slaked fat lime and four times its weight of each sample. These mixtures were all gauged during the same time with 54 per cent of their weight of soft water. The consistency of the pastes was judged by the thicknesses not traversed by the sounding-lead of 300 grammes, and the time taken to set in a moist atmosphere was noticed.

The results were as follow:

Designation of the gaize:	A	B	C	D	E	F	H	K	L
Consistency of the paste	rather soft	good	good	good	rather soft	good	soft	good	good
Thickness not traversed by the sound in mm	5	6	9	12,5	28,5	12	3	7,5	7
Commencement of setting in hours, about	18	18	15	15	(13)?	(12)?	15	20	14
Completion of setting in days, about.	11	11	10	10	10	10	10	11	11

Disregarding paste H, the consistency of which was abnormal, we see that to have pastes of exactly the same consistency, the amount of water used must be greater the finer the powder. Also the finer the samples are ground the quicker is the setting.

Tests of strength were made in two different ways.

1. To approach as nearly as possible to the German method, the mortars were made of one part by weight of powdered lime, two parts of puzzuolana, and 3 parts of silicious sand of rounded grains passing through holes 1·5mm in diameter, but not through holes of 1 mm. With the various samples of gaize it was possible to take the same proportion of water, which was determined by trial to be 1·4 times the weight of lime. The mortars produced looked like damp earth (those made from samples C, D and E, looked a little drier). They were put into briquette-moulds and well rammed with a spatula. The briquettes were taken from the moulds after 24 hours and at once immersed in water, after 28 days they were broken by tension. Each of the results given below is the mean result from six briquettes. They would have been somewhat higher if the mortars had been rammed mechanically.

2. To apply the method proposed, mortars containing by weight 50 parts of lime, 200 of puzzuolana, 750 of the sand above defined, and 180 of water, were gauged with the trowel. All were of a very plastic consistency. They were moulded into prisms measuring 16×4×4 cm, filling the moulds with trowel and finger. When they had set long enough in damp air, the prisms were taken from the moulds, and allowed to further harden in the same air for a week. They then had three weeks in water, and were then broken by bending, making one of the lateral faces bear on two supports 10 cm apart, and exerting a gradually increasing force on the opposite face and midway between the supports. The strength per cm^2 was calculated by the usual formulae, multiplying the total breaking load by 15·64. Each of the results given below is the average given by two prisms.

The halves of the prisms broken by bending were afterwards placed cross-wise between 2 plates of steel 4 cm wide and tested by compression. The resistances obtained were calculated to the cm^2 of the base. Each number is the average of four tests.

Composition of mortar	Nature of mortar	Nature of the test	A	B	C	D	E	F	H	K	L
1 : 2 : 3 : 1,4	beaten dry	Tensile	6,3	6,7	7,3	8,6	10,4	10,9	9,0	6,7	7,2
50 : 200 : 750 : 180	plastic	Bending	6,5	8,1	10,1	12,6	18,0	22,2	15,9	8,8	9,5
		Compression	10,5	11,6	15,6	20,9	29,4	40,7	27,4	14,7	15,0

We see that from A to F, i. e., as the powders become finer, the three sets of resistances increase regularly. The differences are much sharper in the tests with plastic mortar. This shows that this method is more sensitive than the first. As might have been foreseen, the mortar H was distinctly stronger than mortar B, and mortar K was slightly stronger than mortar B. As regards K, the difference was appreciable only with the plastic mortars. Finally, mortar L gave a strength hardly less than mortar C.

2nd Test. A series of analogous tests was made with a mixture of equal weight of three trasses of best quality (yellow, grey and blue) coming from Herfeldt's quarries, and received in blocks. Each trass was first powdered till all of it went through holes 2·5 mm in diameter. Equal parts (by weight) of these powders were mixed and then ground in various ways. We thus got with the ball mill:

Sample	A	B	C	D	E
Number of Revolutions	150	200	500	900	2000

Two samples marked H and K were then made in the same way as the samples of gaize having the same letters in the first test, except that the proportions between the residue from a 4900 sieve, and the fine dust were 32 per cent and 68 per cent, so that they were the same as the sample of trass B.

Finally a sample marked L was obtained by subjecting the material to 350 turns in the ball mill, sifting it through a 4900 sieve after every fifty turns so as only to grind the residue. The final residue was mixed with all the fine powders previously separated. As regards its constituent of residue from the 4900 sieve this mixture approaches sample C. It only differs from C in less prolonged pulverisation of the fine dust.

The sifting and levigation tests executed in the same manner as with the gaize, gave:

Designation of the trass			A	B	C	D	E	H	K	L
Sifting Test	Proportion of grains kept back by a sieve of	4900 meshes	43	32	14,5	9,5	3	32	32	13
		900 „	15,5	7.5	2	1,5	0,5	7,5	0	2,5
		324 „	9	5	1	1	0,2	5	0	1,5
		144 „	5	2	0,5	0,5	0	2	0	0,5
Levigation Test	Proportion of grains not carried away by a current of	2cm p. min.	61,2	60,8	47,5	39,3	31,2	36,0	58,4	49,5
		10 „	46,8	41,5	25.8	14,9	6,1	24,4	43,0	24,9
		30 „	32,6	25,3	3,0	1.6	0,3	20,5	24,9	3,1

Each sample was mixed with a quarter of its weight of the same slaked fat lime powder and the mixtures were all gauged during the same time with 43 per cent by weight of soft water. The resulting pastes were subjected to the same tests as the gaize and with the following result:

Designation of the trass	A	B	C	D	E	H	K	L
Consistency of the pastes	good	good	good	good	rather firm	rather soft	rather soft	good
Thickness not traversed by the sound in mm	5,5	7,5	12	14,5	24	3	4	8.5
Commencement of setting in hours, about	24	24	24	24	24	30	24	16
Completion of setting in days, about	8	8	7	8	7	7	8	7

Excluding the samples H and K, which show the same anomaly as H in the preceding test, all gave (with the sounder) pastes firm in proportion to the degree of fineness to which they had been ground. Nevertheless, with the uniform quantity of water used for gauging we have always obtained pastes of a good plasticity, except with the finest sample E which gave rather too stiff a paste.

The setting tests, difficult with such slow cements, gave nearly the same results with all the samples, or at least differences which do not seem to follow any definite rule.

Tests of strength have been made in the same way as with the gaize; some by tension on dry rammed mortars containing by weight, one of lime, two of trass, three of sand, and 1.1 of water; others by bending followed by compression, using plastic mortars containing by weight 50 of lime, 200 of trass, 750 of sand and 148 of water. In each series, the mortars obtained with the eight samples had practically the same strength. The mean resistances are here given.

Composition of the mortar	Nature of the mortar	Nature of the test	A	B	C	D	E	H	K	L
1 : 2 : 3 : 1,1	rammed dry	tension	12,8	13,9	14,3	14,5	15,8	14,4	12,6	12,8
50 : 200 : 750 : 148	plastic	bending	12,9	14,0	18,2	18,8	21,6	22,5	15,6	15,9
		compression	24.0	24,6	35,4	40,6	48,9	44,5	27,3	28,5

As in the preceding case, the tests with plastic mortar proved more sensitive than those with rammed mortar. In particular they have shown best the differences between samples H and K, and sample B, as well as between L and C.

Conclusion. To sum up, the two series of tests show that the results given by the same puzzuolana, especially the strengths of sand-mortars made with it, vary with its fineness, and depend, not only on the proportion of the residue on a given sieve (for instance the 4900 sieve), but also on the composition of this residue as well as on that of the fine dust. For the results to be always comparable, it is then, at first, necessary that the standard fineness to which the puzzuolanas must be reduced for the characteristic tests should be exactly defined.

The principal object of these tests is to show the energy of which a given puzzuolana is capable, and it is therefore of interest to powder it as finely as possible. But a limit must be set, and, excessive fineness would require a prolonged pulverisation often difficult of realisation in laboratories. Besides, a definite composition could not be prescribed, as it is almost impossible to obtain it exactly, and it is therefore necessary to allow a certain margin. According to the preceding experiments, a difference of 10 per cent in the amount of residue on a 4900 sieve involves appreciable

difference in resistance, and should not be exceeded. One might then, at first, allow a residue of 25 per cent on that sieve, so as to get nearly the same fineness as for cements, with a margin of 5 per cent either way.

This fixed, it seems impractical to define the compositions of the residue and of the fine powder, by fixing the amount of residue which the powder ought to leave on other sieves or in a levigation test, since a set of numbers which might suit for one puzzoulana might be unattainable (except by mixing and eliminating) with a puzzuolana different from the first in nature and hardness. It would be preferable to decide on a method of pulverisation; for instance by prescribing that the puzzuolana should be pulverized without any sifting during the process, until the amount failing to pass the 4900 sieve comes within the limits just proposed. No doubt the compositions obtained will vary somewhat according to the kind of mill used, but the differences should be unimportant, and it would complicate the tests too much to insist upon any particular form of mill.

These conditions apply exclusively to the standard tests (called above «characteristic») and destined for the study of various puzzuolanas under invariable conditions. When it is a question of testing a puzzuolana in the state of fineness in which it is to be used in the yard, then the matter comes within the province of the «practical» tests discussed later, and it will be sufficient to define the granulometric composition by the tests discussed in chapter L.

B. Influence of the Lime.

The tests were made with four different limes, some slaked sifted, or preserved in various ways.

Except for samples G and J, which will be discussed later, the lime was slaked by putting the quick lime in pieces in a wooden box, and watering it with rather less water than was necessary to convert it entirely into hydrate. The lime was then covered up and left for two or three days in a cellar. During this time the slaking took place and the lime fell to dust. It was then put either through a sieve with 144 meshes to the cm^2, or more usually through one of 900 meshes. The sifted powder was

gathered and will be called «lime of first slaking». The residue was returned to the box, was again watered, and then left rather longer than before to slake. When sifted, this lime is the «lime of second slaking». With one of the limes a third slaking was carried out, and the remainder of the lime was then thrown away.

A first quantity of quicklime coming from Etaples near Boulogne gave the following samples:

A) Lime of first slaking passed through a 900 sieve, and then kept in an air-tight box for a month before testing.

B) Lime of first slaking, passed through a 144 sieve, and then kept like A.

C) Lime of third slaking, passed through a 900 sieve, and then kept like A.

D) Lime resulting from a first slaking during which watered pieces remained for about a month in the box and were then passed through a 900 sieve a few days before being used.

E) Lime obtained as in D but passed through the 144 sieve, instead of through the 900 sieve.

F) Lime of first slaking passed through the 900 sieve (the same as A), then exposed to damp air for a month in the state of powder.

G) Pieces of quicklime from the same supply were placed with water in an autoclave, which was at once hermetically closed and then heated for about one day up to a pressure of 2 to 3 atms. The excess of vapour was then allowed to escape, and, after cooling, the slaked lime was passed through a sieve of 900 meshes to the cm^2. Before being tested, the powder was keptfor about one month in an air-tight box.

Another quantity of lime from the same place, but previously supplied, was also used, by mixing limes of first and second slaking, and sifting it through the 900 sieve; this sample has been denoted by H; when tested, it had been kept for about 2 years and a half in an air-tight box, which however had been opened from time to time, to take out what was wanted for use.

A third lime, somewhat less pure, and kept for a long time as blocks in a sack, was gradually slaked by the dampness of the atmosphere, and the greater part of it had fallen into powder.

Shortly before being tested, it was passed through the 900 sieve, and the fine powder thus obtained was denoted by J.

Finally a fat lime from Liège (Belgium) was tested and compared with the preceding ones, according to the express desire of Mr. G. Herfeldt, President of the Commission No. 11. This lime was received in lumps as quicklime and furnished two samples.

K) Lime of first slaking, passed through the 900 sieve and then kept for about six months in a well closed box, until it was to be tested.

L) The residue from this first sifting was slaked again, but with too much water. After 15 days slaking, the lime was dried on a sand bath, to get rid of the excess of water, was then passed through a 900 sieve, and the powder was kept in a well closed box for 6 months before the tests.

The chemical analysis of these limes gave the following results.

Sample of lime	A	B	C	D	E	F	G	H	J	K	L
Monohydrated lime .	86,99	86,21	92,72	—	89,87	—	—	88,06	66,15	95,14	—
Monohydr. magnesia .	0,46	0,42	0,45	—	0,45	--	—	0.29	0,68	0,42	—
Carbonate of lime . .	4,43	4,66	3,75	5,57	6,59	28,19	3,18	9,09	5,45	3,18	9,43
Sulphate of lime . . .	0,76	0,86	1,27	—	0,97	—	—	0,54	0,93	0,53	—
Silica	0,91	0,97	0,79	—	1,09	—	—	0,92	8,72	0,40	—
Alumina	0,59	0,57	0,77	—	0,73	—	—	0,47	3,29	0,15	--
Oxide of iron	0,13	0,16	0,25	—	0,23	—	—	0.11	1,16	0,05	—
Unhydrated lime . . . (quick or combined with SiO_2, Al_2O_3 or Fe_2O_3)	5,73	6,15	0	—	0	—	—	0	13,62	0	—
Water in excess . . .	0	0	0	—	0,07	—	—	0,52	0	0.13	—

As will be seen all the limes depart from the limits proposed a priori for the definition of a standard fat lime. The nearest to it is sample K (Liège lime, first slaking) which contains rather less than 5 per cent of impurities. These different limes were tried comparatively with the four following puzzuolanas, each being finely ground.

	Residue on the 4900 sieve.
Gaize slightly burnt (different from that of 1st tests A)	22 per cent
Trass (different from that of 2nd tests A)	16 „ „
Blast furnace slag (quenched)	32 „ „
Roman puzzuolana.	27 „ „

We first mixed lime with four times its weight of puzzuolana, and the same quantity of water for each sort of lime, to pastes without sand, which were tested like those of the last chapter with the sound and with the Vicat needle. The results are as follows:

Gaize-pastes all gauged with 53 per cent of water.

Designation of the lime:	A	D	E	G	H	J	K
Consistency of the paste:	good	good	good	rather stiff	good	good	good
Thickness not traversed by the sound in mm	8,5	7,5	9	16,5	7,5	10,5	13,5
Beginning of setting, in hours (about)	15	21	22	20	20	15	17
End of setting, in days (about)	8	8	8	8	8	8	8

Trass-pastes all gauged with 40 per cent of water:

Designation of the lime:	A	D	E	G	H	J	K
Consistency of the paste:	good	good	rather soft	rather stiff	good	good	good
Thickness not traversed by the sound in mm	9,5	7	6,5	(?)	12	11	8
End of setting, in days (about)	7	7	8	8	$7^1/_2$	7	6

The beginning of the setting was too uncertain to be accurately noticed.

Slag-pastes all gauged with 30 per cent of water.

Designation of the lime:	A	D	E	G	H	J	K
Consistency of the paste:	good	good	good	rather stiff	good	good	good
Thickness not traversed by the sound in mm	9,5	6,5	11	12	6	10,5	11
Beginning of setting, in hours (about)	2	2	1	4	2	1	2
End of setting, in hours (about)	51	64	64	64	64	64	51

No pastes without sand were made with the Roman puzzuolana.

These three sets of tests show that the different sorts of lime required the same quantity of water to give pastes of good plastic consistency with the same puzzuolana. Lime G, slaked in the digester, however, required rather more water.

As to the setting-tests, which are very uncertain, they show nothing very clearly. It would seem, however, that lime J generally sets quicker than the rest, which might have been expected from its greater percentage of hydraulic substances.

Besides these tests, we have filled needle-cylinders (Le Chatelier's system) for expansion tests both with pastes of gaize and of trass. Immediately after gauging, a small plate of glass was put on the two bases of the cylinders, and they were immersed in soft water. After a week's immersion the plates of glass were removed and the distances between the points of the needles were measured. The cylinders were then re-immersed. Three weeks later, the distances were again measured. The cylinders were then put into water which was so heated that it boiled in half an hour, and kept boiling for three hours. After cooling, the distances were again measured.

During the three weeks in cold water, the increase in the distances between the needle points was about 1 mm, for all pastes. During the subsequent boiling, it increased from one to three mm. The various limes, therefore, behaved in the same way. The one which gave the greatest expansion (3 mm on boiling with the trass, and 2 mm on boiling with the gaize) is lime E, which had been passed through the 144 sieve.

Comparative tests of resistance were made in the same way as those in chapter A, on the one hand by tension on rammed mortars 1 : 2 : 3 made with the various limes and each puzzuolana, and on the other hand by bending, followed by compression, using plastic mortars 50 : 200 : 750. For each series of mortars, the same proportion of water was used, determined by preliminary trials. The results obtained with the four puzzuolanas are as follows:

Composition of the mortar	Nature of the mortar	Lime:	A	B	C	D	E	F	G	H	J	K	L
Gaize slightly burnt													
1 : 2 : 3 : 1,43	ammed dry	Consistency	good	good	good	good	good	good	rather dry	good	rather dry	good	rather dry
		Tension	7,4	8,5	7,1	7,4	7,6	7,5	9,2	8,1	10,7	9,1	8,5
50 : 200 : 750 : 185	plastic	Consistency	good	good	good	good	good	good	good	good	good	good	good
		Bending	11,9	12,4	10,3	10,7	11,1	10,6	11,8	13,8	12,3	14,4	14,6
		Compression	19,6	19,1	17,9	18,1	17,7	16,7	18,0	22,4	20,1	22,9	22,9
Trass													
1 : 2 : 3 : 1,10	rammed dry	Consistency	good			good	good			good	rather dry	good	
		Tension	11,6			12,6	13,3			12,8	13,5	11,8	
50 : 200 : 750 : 155	plastic	Consistency	good			good	good			good	good	good	
		Bending	17,7			16,8	16,8			17,9	16,4	20,2	
		Compression	45,3			42,5	43,7			46,5	41,0	52,7	
Blast furnace slag (quenched)													
1 : 2 : 3 : 0,93	rammed dry	Consistency	good	good	good	good	good	good	rather dry	good	rather dry	good	rather dry
		Tension	16,4	15,8	17,0	17,2	16,6	19,0	14,3	19,5	18,3	16,5	20,1
50 : 200 : 750 : 140	plastic	Consistency	good	good	good	good	good	good	good	good	good[1])	good[2])	good
		Bending	18,8	19,6	18,5	18,5	17,7	18,0	20,5	18,0	14,7[1])	21,7[2])	20,2
		Compression	52,7	53,9	51,9	48,0	49,6	44,1	56,1	48,1	39,4[1])	66,0[2])	55,8
Roman puzzuolana													
1 : 2 : 3 : 1,00	rammed dry	Consistency	good		good	good	good	good	rather dry	good	rather dry	good	rather dry
		Tension	10,7		11,3	10,8	11,5	10,6	9,6	12,3	12,9	11,2	13,7
50 : 200 : 750 : 135	plastic	Consistency	good		good	good	good	good	good	good	good	good	good
		Bending	14,6		14,3	13,5	14,3	15,2	14,6	14,6	13,8	15,8	16,4
		Compression	40,3		39 0	41,4	40,2	40,3	41,3	39,1	41,0	41,4	43,3

[1]) Gauged by mistake with 150 of water instead of 140.

[2]) Gauged by mistake with 132 of water instead of 140.

We see that with the same puzzuolana all the limes required practically the same amount of water to give mortars of the same consistency. The limes G, J, and L, alone wanted rather more water in the rammed mortars. All the plastic mortars had the same consistency.

For the resistances the relative differences are better shown by the following table, compiled to bring the resistances of the mortars to percentages calculated to lime A as standard.

	Lime:	A	B	C	D	E	F	G	H	J	K	L
Rammed Mortars Tension	Gaize	100	105	96	100	103	102	124	110	145	123	115
	Trass	100	—	—	109	115	—	—	110	116	102	—
	Slag	100	96	104	105	101	116	87	119	112	101	122
	Rome	100	—	106	101	107	99	90	115	121	105	128
	Average	100	100	102	104	106	106	100	113	123	108	122
Plastic Mortars Bending	Gaize	100	104	87	90	93	89	99	116	103	121	123
	Trass	100	—	—	95	95	—	—	101	93	114	—
	Slag	100	104	99	99	94	96	109	96	78[1])	116[2])	108
	Rome	100	—	98	92	98	104	100	100	95	108	112
	Average	100	104	95	94	95	96	103	103	97	114	114
Plastic Mortars Compression	Gaize	100	97	91	92	90	85	92	114	102	117	117
	Trass	100	—	—	94	97	—	—	103	91	116	—
	Slag	100	102	98	91	94	84	103	91	75[1])	125[2])	106
	Rome	100	—	97	103	100	100	102	97	102	103	107
	Average	100	100	95	95	95	90	99	101	96	110	110

[1]) Gauged by mistake with 150 of water instead of 140
[2]) Gauged by mistake with 132 of water instead of 140
} not reckoned in the averages.

This table shows that the influence of the method of slaking the lime and of its fineness (whether the sieve had 900 or 144 meshes) is insignificant, and also that the lime may absorb a rather large amount of carbonic acid without the strength of the mortar being sensibly changed. The chemical composition has an effect rather more marked, although very small. The fourth lime alone (samples K and L) which was the purest, always gave a greater resistance than the first in the three series of tests.

We may also remark that the bending and compression tests on plastic mortars have generally given consistent results and in close agreement with those which might have been expected, taking into consideration the differences in composition or preparation between the limes. On the contrary, the tensile tests on dry rammed mortars have often given improbable results: thus the limes somewhat inert, *D*, *E*, *F*, *H*, on the one hand, and *L* on the other, have given higher resistances than the fresh limes of the same composition *A* and *K*; the impure and inert lime *J* had a greater resistance than the purer limes *A*, *H* and *K*.

Conclusions. The standard fat limes to be used in the tests need not be defined so rigorously as might have been supposed.

It is useless to prescribe any particular method of slaking, provided the slaking of the sifted powder is complete. This is secured by taking the proportion of water to free lime not less than 24·3 per cent. On the other hand, 30 might be taken as the maximum.

It seems immaterial whether the sifting is done with sieves of 900 or of 144 meshes to the cm^2. Nevertheless the 900 sieve is preferable, as there is always the risk that with very recently slaked limes the larger grains are insufficiently hydrated.

Finally, the maximum amount of impurities in the lime might be fixed at 3 per cent, excluding, however, carbonate of lime, which latter might be allowed to reach 7 per cent.

C. Influence of the Sand.

The following sands have been compared:

A) Ground marble in angular grains containing 2 per cent of substances insoluble in hydrochloric acid.

B) Shelly beach sand, containing 19 per cent of matter insoluble in the acid.

C) A mixture of various sands, rather shelly, containing 57 per cent insoluble matter.

D) Seine sand in rounded grains, containing 80 per cent insoluble matter.

E) Leucate (Aude) sand in rounded grains, of which 96 per cent was insoluble.

F) Cherbourg quartzite, ground into angular grains, entirely silicious and insoluble.

G) Freienwalde sand, from which the standard German sand is got, in rounded grains, silicious and quite insoluble in the acid.

These sands have been graded into various sizes by means of sieves with circular holes 2, 1·5, 1·0 and 0·5 mm, in diameter.

With the same fat lime and the same Roman puzzuolana, finely ground (24,5 per cent of it remaining on a sieve of 4900 meshes to the cm^2), we made, as in the preceding experiments, a series of dry rammed mortars in the proportion of 1 : 2 : 3 for tensile tests, and a plastic series in the proportion of 1 : 4 : 15 for bending and compression tests.

For the dry mortar, sand which passed holes of 1·5 mm and was retained by holes of one mm was used exclusively.

For the plastic mortars, three sizes of grains were mixed in equal weights, the grades being 2—1,5, 1,5—1,0, and 1,0—0,5. Another plastic mortar *B'* was made with beach sand passing 2 mm holes and retained by 0,5 mm holes and in which the proportions between the three sizes of grains were 39, 43, and 18 per cent, and also a plastic mortar *G'* of Freienwalde sand composed of 1,5—1·0 grains and of 1,0—0,5 grains mixed in equal portions.

The requisite quantity of water for each mortar was ascertained by experiment.

The test pieces were all made and immersed under the same conditions as in the preceding tests, and broken after 28 days. The only difference is that the bending tests were so executed that the prism placed vertically was subjected to a constant bending stress on a part of its length, instead of being supported at the ends and loaded in the centre.

Composition of mortar		Sand	A	B	C	D	E	F	G	B'	G'
1 : 2 : 3	Dry rammed mortars	Mixing water	1,10	1,06	1,07	1,00	0,96	0,99	0,93	—	—
		Resistance to tension per cm^2	13,3	12,1	12,3	13,1	13,7	12,8	14,1	—	—
		Proportional numbers	97	88	90	96	100	93	103	—	—
50:200:750	Plastic mortars	Mixing water	160	140	140	135	125	145	130	135	135
		Resistance to bending per cm^2	16,4	13,5	14,4	14,1	15,1	9,2	7,9	14,5	7,4
		Proportional numbers	109	89	95	93	100	61	52	96	49
		Resistance to compression per cm^2	43,5	37,6	42,7	41,1	43,6	22,7	26,0	43,9	23,0
		Proportional numbers	100	86	98	94	100	52	60	101	53

The most striking result is the weakness of the plastic mortar made with the sand F, G, and G' consisting of nearly pure silica.

It will be advisable to adopt for testing puzzuolanas the same standard sand as for cements, and the definition of this sand is now under the consideration of a special commission.

D. Choice of the proportions of lime, sand, and puzzuolana.

Preliminary tests, some of which have been described in another report*), show that among all the cements which can be made by mixing a powdered fat slaked lime with a finely powdered puzzuolana, the mixture which gives the strongest mortars contains in general about four parts of puzzuolana to one of lime (with quenched blast furnace slags less lime must be taken). It is therefore advantageous to adopt that proportion in making tests. As to the proportion of sand, it is quite natural to take a quantity equal to three times the quantity of binding material, as in the cement tests. We thus arrive at the composition 1 : 4 : 15 or 50 : 200 : 750, which was employed for all the plastic mortars above considered.

The following tests were made with cements composed respectively of 5, 4, and 3 parts of puzzuolana to one of lime, and mixed with two or three times their weight of sand. The mixing water was determined by trial with one kg of dry mixture, so as to give always very plastic mortars. The tests were carried on exactly as the preceding ones and were limited to 28 days. In the bending tests the prisms were broken by applying the load in the centre.

As might have been expected, the mortars with two parts of sand were stronger than those with three, but in all cases the strength is sufficiently great with the principal kinds of puzzuolana for the latter composition to be adhered to.

*) International Congress of Methods of testing building materials, held at Paris in 1900, Vol. 11, part 2, p. 104.

Weight of puzzuolana to one of lime		5	4	3	5	4	3
Weight of sand to one of the mixture of lime and puzzuolana		2	2	2	3	3	3
Composition of the whole mixture		1:5:12	1:4:10	1:3:8	1:5:18	1:4:15	1:3:12
Crushed Bricks	Mixing water	168	173	175	147	152	155
	Bending	2,0	1,9	1,7	1,2	1,0	1,4
	Compression	5,3	5,0	4,7	3,8	3,5	3,8
Baked Gaize	Mixing water	200	200	210	170	174	177
	Bending	10,3	8,9	6,2	7,4	6,2	5,9
	Compression	16,6	15,6	10,3	12,4	10,9	10,3
Trass	Mixing water	160	165	172	142	147	153
	Bending	18,2	17,3	16,8	13,8	14,0	10,4
	Compression	47	43	43	36	37	31
Roman Puzzuolana	Mixing water	148	155	160	134	140	141
	Bending	23,4	20,5	20,5	17,1	15,3	16,5
	Compression	59	50	51	37	38	45
Slag	Mixing water	146	144	156	130	134	141
	Bending	27,8	25,5	25,2	23,4	22,5	19,4
	Compression	89	84	77	63	56	52
Another Slag	Mixing water	153	154	158	135	138	146
	Bending	40,8	36,9	31,1	28,7	26,7	24,6
	Compression	137	130	118	94	95	76

With the mortars with two parts of sand the strength always increased with the proportion of puzzuolana, but in the case of those with three parts of sand, they varied sometimes one way, sometimes the other; and it seems that the proportion 1 : 4 between lime and puzzuolana represents a good average.

Hence there is no objection to adopting the proportions 1:4:15, the choice of which has been mentioned above.

We shall see later, in chapter N, the report of tests in which the proportions of the three constituents were varied far outside these limits.

E. Method of mixing.

In all the tests here reported, the rules for mixing pastes without sand, or plastic mortars with sand, have been followed, and are indicated in the programme of experiments circulated in 1902. It has thus again been shown that nothing can be urged against these rules, and that there is no reason for making any change in those proposals.

F. Proportion of water for the standard paste.

We have seen from the tests described in chapters A and B, that in general with pastes of a consistency regarded as good, the sounding rod 10 mm in diameter and weighing 300 grammes stopped at a distance of from 5 mm to about 14 mm from the bottom of the box. Taking the mean of these distances which were not penetrated by the rod in the 28 pastes of good consistency, and in a few other similar trials, the distance is reckoned at about 9 mm. But if we take account only of puzzuolanas leaving 20—30 per cent on the 4900 sieve, this average comes down to 8 mm, a greater thickness than the 6 mm considered suitable for cements.

From the variety in practice in pastes of good consistency, it is probable that if we define the standard consistency of pastes made with puzzuolanas of standard fineness by a constant thickness, such as 8 mm, untraversed by the sounding rod, we should arrive at somewhat different consistencies with different puzzuolanas. But that would be of secondary importance if the differences never exceeded a certain limit, provided that the rule adopted ensured easily comparable conditions of work.

The experiments tabulated below show the sensitiveness of the method when it is in the hands of the same operator. The difficulty lies in defining with sufficient precision the method of mixing and the method of filling the box, so as ensure that the sounding rod, when used by different operators, shall sink to the same depth for the same proportion of water.

Puzzuolana	Proportion retained by the 4900 sieve	Water added per kg of the mixture	Consistency	Thickness not traversed by the sounding rod
Baked gaize	27 per cent	545 grammes	rather soft	4 mm
		525 »	good	6,5 »
Roman puzzuolana . .	24,5 » »	390 grammes	rather soft	3 »
		375 »	good	5,5 »
		365 »	rather stiff	13 »
Santorin earth	15,5 » »	435 grammes	too soft	5,5 »
		430 »	rather soft	7 »
		422 »	good	11 »

The question is the same as in the case of pastes of pure cement, and would be resolved together, so that the same method may be adopted in both cases, except perhaps that the figure decided on for the thickness untraversed by the sounding rod might be modified.

There would, besides, not be more to take into account if, as we propose later in chapter H, it is decided that the setting tests of puzzuolana should be made with a standard sand-mortar.

G. Proportion of water for standard plastic mortars.

By reason of the difficulty met with in defining the standard consistency of sandless pastes, it need not be attempted at present to deduce from the corresponding proportion of water consistency of standard sand-mortars made with the same puzzuolanas. Moreover, the coefficients of the formula to be used would depend, of necessity, upon the nature of the sand adopted as the standard, and this has yet to be fixed. Finally if it is decided not to institute setting tests with sandless pastes, we need not trouble about them further, and the consistency of standard sand-mortars shall be defined without them.

But in the meantime, it is important to discover what effect differences in the quantity of the water added may have on the strength of puzzuolana mortars. With this object we tested as

above dry rammed mortars (1 : 2 : 3) by tension, and also plastic mortars (50 : 200 : 750) by bending, with a constant moment, and finally by compression.

We used a Roman puzzuolana powder leaving 24,5 per cent on the 4900 sieve.

The rammed mortars gave:

Weight of water to one of lime	0,84	0,90	0,93	0,96	0,99	1,02
Consistency [1])	too dry	rather dry	good	good	rather soft	too soft
Resistance to tension per cm²	13,3	12,7	12,7	12,3	12,0	12,2

The plastic mortars were much affected by variations in the water, the strength decreasing faster as the proportion of water increased.

Weight of water to one kg of mixture	120 gms.	130 gms.	135 gms.	140 gms.	150 gms.
Consistency [2])	too stiff	rather stiff	good	rather soft	too soft
Resistance to bending per cm² . . .	16,9	15,4	14,4	13,6	11,5
Resistance to compression per cm²	56,2	45,3	41,1	36,3	29,4

To make the results comparable, it is thus indispensable to define exactly how much water is to be used for mixing.

The rules for testing trass, adopted in Germany, direct that from 0,9 to 1,0 of water should be taken for one part by weight of lime, 2 of trass, and 3 of sand, thus allowing a margin of one tenth of the weight of the lime; i. e of 17 gms. of water per kg of dry mixture. This is too much, having regard to the corresponding variations in resistance, and yet it would be necessary to allow even more latitude if we wished the rule to be applicable to all puzzuolanas. We have seen, in fact, by the examples already given in this report that, according to the puzzuolana used in rammed mortars of 1 : 2 : 3, the quantity of water giving the best consistency varies from 0,93 to 1,43.

[1]) The consistency considered good is the dry consistency most suitable for ramming into the moulds.

[2]) The consistency considered good was a very plastic one.

It is necessary therefore in the case of the proposed plastic mortars that the proportion of water to be used should be defined much more precisely.

With the object of fixing a method permitting the realisation of this, we have made many experiments with the sounding rod loaded up to one kg, but the depths to which it penetrated were observed to be different. But when the sounding rod was loaded till it weighed 2 kg, we got numbers much more in accord. The results with Leucate sand mortars 50 : 200 : 750, were as follow:

Puzzuolana	per cent not passing the 4900 sieve	Mixing water in gms. per kg of dry mixture	Consistency	Thickness not traversed by 2 kg sound. rod
Baked kaolin .	2 (?)	130	rather stiff	24,5
		140	good	6,5
		150	rather soft	5
Slag.	11,5	115	rather stiff	12,5
		125	good	6
		135	soft	3,5
Another slag. .	20,5	110	rather stiff	12
		120	good	6
		130	soft	3
Baked gaize . .	27	152	rather stiff	21,5
		162	good	6,5
		172	rather soft	4
Roman puzzuolana . . .	27	105	stiff	30
		115	good	7
		125	soft	3,5
Artificial puzzuolana (a sort of slag)	27,5	105	rather stiff	11,5
		115	good	6
		125	soft	3,5
Trass.	29·5	125	rather stiff	18,5
		135	good	5,5
		145	soft	3
Another trass .	50	130	rather stiff	24
		140	good	7
		150	rather soft	4,5

In spite of the differences in fineness in the puzzuolanas tested, the thicknesses not penetrated in mortars of good plastic consistency were always about 6 mm.

Besides, the fact that the differences between these figures and those found with mortars rather too stiff or rather too soft are very considerable, shows the great sensitiveness of the method.

We might thus define the standard consistency of mortars of standard composition as one in which the 2 kg sounding rod, used like the 300 gms. sounding rod for cements stops 6 mm from the bottom of the box[1]).

This number might however have to be modified later, according to the size and form of the grains of the standard sand, when that has been settled by the International Association.

H. Setting Tests.

By reason of the fineness of the subdivision of the proposed standard puzzuolana, pastes of lime and puzzuolana may be subjected to the same setting tests as pure cements, and it seems useless to make any special rules.

Nevertheless, the method currently adopted for cements is open to various criticisms, especially as it is difficult to fix exactly on the time of completion of the setting; and a special committee of the International Associacion is charged with the duty of studying what modification should be made to make the test more precise. With pastes of fat lime and puzzuolana, which generally set very slowly, this difficulty is even greater than with cements, and we have already seen, in chapters A and B, examples of results which disagree owing to the insufficient delicacy of the method.

The German rules for testing trass direct the determination, after 2, 3, 4 and 5 days, of the force necessary to drive a round needle of one mm^2 in section right through the paste. This method is more delicate even than the ordinary method, for it requires an estimate, not only of the cessation of the penetration of the needle, but of the load on the needle at that exact instant.

[1]) This method is not applicable to cement-mortars.

In our search for greater precision, we have made various experiments assuming that the setting is finished when a Vicat needle 5 kg in weight penetrates 5 mm into the paste by its own weight only. With this object we have subjected the same mass of paste to three sets of tests:

1. The penetration of the ordinary 300 grms. needle was measured after succesive intervals of time.

2. The same was done with the needle loaded to weigh 5 kg.

3. Having placed the box of paste on a spring balance and taken its weight, a round needle of one mm^2 section was pushed into the paste by hand. 5 mm from the point of the needle was a large shoulder, and the balance was read at the instant this touched the paste. The difference between the two readings of the balance shows the force required to drive the needle in 5 mm.

Taking as abscissae the times of the testing reckoned from the mixing, and as ordinates on one hand the penetration measured in the second set of tests, and on the other the results of the third set, and then joining by continuous lines the two series of points, we get two curves which ought to be such that the same abscissae would correspond to the 5 mm ordinate of the first and the 5 kg ordinate of the second. On the other hand the length of this abscissae relative to that of the completion of the setting as determined in the first set would show whether the period denoted by the abscissae can be taken to denote with any degree of accuracy the actual termination of the setting.

Such experiments have been repeated with a large number of pastes, including all those mentioned in chapters A and B, but the two abscissae rarely coincided, and sometimes they showed differences of more than a day. Moreover their mean value has almost always, especially with the slowest setting puzzuolanas, corresponded to a longer period than that after which the 300 gms. needle ceased to make any perceptible impression on the surface of the paste.

This latter point would be of small importance, since the end of the setting is a purely conventional moment in the continual hardening of mortars, and can be put sooner or later without inconvenience. It is simply a matter of definition. But the disagreement between the results supplied by sets 2 and 3,

proves that one set at least is defective. Besides, it would be of little use to join points by a curve as we have done, and it would after all be necessary to determine at hap-hazard, either the instant at which penetration is exactly 5 mm and no more under a load of 5 kg, or, that at which the force necessary to give that penetration is exactly 5 kg. Both methods would then be subject to uncertainties of the same kind as those to which the present method is subject.

In view of these difficulties it might be supposed that results could be obtained by the old method which would be quite as approximate, and possibly even more satisfactory. This consisted in watching for the instant at which the paste ceased to yield to the pressure of the thumb. This method combines extreme simplicity with the great advantage of being equally applicable to sand-mortars, so that all the characteristic tests of puzzuolanas could be done with the standard sand mortar, while it would be unnecessary to fix upon a standard paste without sand.

To discover the degree of precision of which this method was capable, we made the following experiments with plastic mortars, 1 : 4 : 15, made with the same fat lime, but with various puzzuolanas, and with 2·0—0·5 Leucate sand of the composition already given, and with the water necessary to get a good plastic consistency. For each puzzuolana 2 kg of dry mixture were watered at a time, and the mortar was put into five boxes of the usual type for ordinary setting tests. These boxes were numbered and kept together. Each was put under the surveillance of a different operator, and they were required to note (without communicating their observations to one another) the time at which they judged that a thumb ceased to make any visible impression.

The following table gives the number of hours calculated by each operator, the means of those numbers, the relative differences between individual observations and the mean of the periods necessary for setting. The numbers in brackets denote that the setting was completed during the night, and for these we have taken the mean between the last evening observation, and the first taken next morning. The error made in each of these cases cannot exceed 5 or 6 hours.

Observer:	Time in hours taken to set						Percentage relative differences from the means						Resistance per cm² to compression after the mean setting time
	individual observations					Averages	individual observations					Mean disregarding signs.	
	A	B	C	D	E		A	B	C	D	E		
							p. 100	p. 100	p. 100	p. 100	p. 100	p. 100	kg.
Trass	152	176	191	(184)	172	175	— 13,2	+ 0,6	+ 9,1	(+ 5,1)	— 1,7	8,2	5,6
Baked kaolin	98	119	141	113	141	122	— 19,5	— 2,4	+ 15,6	— 7,4	+ 15,6	12,1	6,1
Artifical puzzuolana . .	98	117	(104)	(104)	121	109	— 10,1	+ 7,3	(— 4,6)	(— 4,6)	+ 11,0	7,5	6,1
Roman puzzuolana . .	91	(105)	98	113	120	105	— 13,4	(0)	— 6,7	+ 7,6	+ 14,3	8,4	5,1
Baked gaize	94	96	97	97	97	96	— 2,1	0	+ 1,0	+ 1,0	+ 1,0	1,0	4,8
Slag	73	(79)	(79)	(79)	73	77	— 5,2	(+ 2,6)	(+ 2,6)	(+ 2,6)	— 5,2	3,6	7,0
Another slag	29	(35)	(35)	(35)	46	36	— 19,4	(— 2,8)	(— 2,8)	(— 2,8)	+ 27,8	11,1	6,5
Portland cement [1]) . . .	(34)	(34)	41	44	41	39	(— 12,8)	(— 12,8)	+ 5,1	+ 12,8	+ 5,1	8,7	4,3
Another cement [1]) . . .	26	27	23	(32)	23	26	0	+ 3,8	— 11,5	(+ 23,0)	— 11,5	10,0	4,6
Roman cement [1]) (Minutes)	45′	43′	60′	50′	60′	52′	— 13,5	— 17,3	+ 15,4	— 3,8	+ 15,4	13,1	6,3
						Mean	— 10,9	— 2,1	+ 2,1	+ 3,3	+ 7,2	8,4	5,6

[1]) In these groups of experiments the Leucate sand was thrice the weight of the cement.

The mean differences thus barely reach 8 per cent of the setting times, which is little compared with the differences between the setting times for mortars made with various puzzuolanas. Now, the method permits of classifying puzzuolanas with sufficient accuracy, and deserves from its simplicity to be recommended until a more accurate one appears.

In a second series of tests we moulded, with mortars of exactly the same composition, cubic blocks of which the resistance to compression was measured at the end of the mean setting periods derived from the first tests. The results of the compression tests are shown in the last column of the last table. They vary little, especially considering that the strongest of them correspond generally to mortars, which, according to new observations by the same five operators, must have become rather harder than in the first test. We may then admit, with the proportions and the kind of sand concerned, that a degree of setting corresponding to the cessation of impressibility by the thumb, means a resistance to compression of 5 kg per cm^2. This would admit of the use of a series of compression tests as a criterion of setting, whereby the time at which the resistance was 5 kg per cm^2, and at which therefore the setting was complete, could be determined by interpolation, or the compression could be tried once at the end of a fixed period. With prismatic test pieces 4 cm^2 the total force required would usually be about 80 kg, and might be exerted by a very simple lever apparatus.

Nevertheless we consider it preferable to retain the much more practical thumb test, and only occasionally to make compression tests to control and educate the operator, or as a final test in cases of dispute.

For current experiments it would be well to operate on one kg of the dry mixture and to fill two boxes with the mortar, one box being used for the preliminary trial tests, and the other being reserved intact for the final tests to be carried out when the thumb only makes a light impression.

I. Resistance Tests.

Most of the experiments already described have shown that the tests of plastic mortars by bending and compression brought out the differences in quality between puzzuolanas better than tensile

tests did in the case of dried rammed mortars. Hence the decision to substitute plastic for rammed mortars, at which Commission No. 11 arrived at its meeting in Budapest, has been fully justified.

At the same time these experiments show that the strength of good puzzuolanas in plastic mortars, is sufficiently high to preclude the idea that the differences in their resistances are not of the same magnitude as errors in experiment. In this connection the resistances of such mortars to tension, which amount to barely half the resistance to bending, would give less clear indications than the latter, and a fortiori than compression tests. The bending tests have other advantages over tensile tests; the form of the test pieces is simple and easily defined and verified, and an apparatus to break upright prisms under a constant moment is also much simpler than the double lever machine used for tensile tests. Finally, the strength deduced from the bending tests approximates more nearly to the real strength than that deduced from the results of the tensile test.

In our programme of experiments of 1902, we have explained the advantages of breaking by constant moment as compared with a central load, and in an appendix to the programme[1]) we have described an apparatus, represented by fig. 1, by which this kind of rupture can be easily effected.

Since then we have made a great number of tests of this apparatus in order to determine the best method of using it. It would take too long to describe them here, and, besides, they are not quite complete. They may one day be the subject of a special report, and some of them have been described in a communication to the meeting on October 25th 1902, by the French and Belgian members of the International Association. At present we are in a position to affirm that the apparatus is very practical. We shall see later that it gives very concordant results. The only point which remains to be decided is the length to give to the prism, or, which is the same thing, the value to adopt for the vertical distance AA' between the inner gripping points, between which the bending moment is the same at all sections. We propose to fix the length of the prisms provisionally at 16 cm, so that 2 prisms

[1]) Les matériaux de construction, 7th year (1902), No. 7., p. 105, and No. 15 p. 235. Baumaterialkunde, Jahrgang VII (1902) Heft 13, S. 207, and Heft 15, S. 235.

can be made with one kg of dry mixture, and to make the distance between the gripping points equal to 4 cm. This arrangement, adopted in most of our tests, has given satisfactory results.

As regards the resistances to compression, we have nothing to add to what has been said in the programme of experiments, viz. that the test on half prisms of 4 × 4 cm crushed crosswise between two steel plates 4 cm wide gives the same resistances

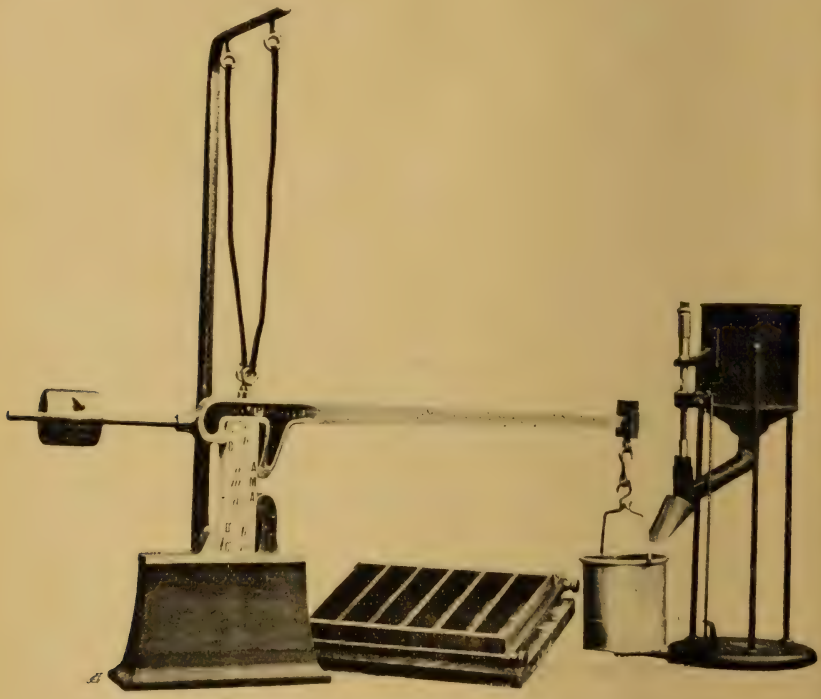


Fig. 1.

per cm^2 as the tests with cubes of 4 or 7 cm side. Fig. 2 gives a proof of this. This represents results of tests on a large number of mortars, some of lime and puzzuolana, others of cement, of various ages and composition, tested by compression both in prisms and in cubes. Each point refers to a different mortar, and the abscissae represent the resistance deduced from tests of cubes (round points refer to cubes of 7 cm, and crosses to those of 4 cm and the ordinate represents that deduced from prisms of 4 cm

side previously broken by bending. It is shown that in general the points representing the various mortars differ little from the bisector of the axes of the co-ordinates.

One of the fundamental points to be examined in fixing upon any method of testing is the degree of concordance in the results which it furnished with test pieces as nearly identical as possible. We have done it with five different puzzuolanas for the bending and compression tests on plastic mortars, as well as for tensile tests of rammed mortars. In the three cases we have

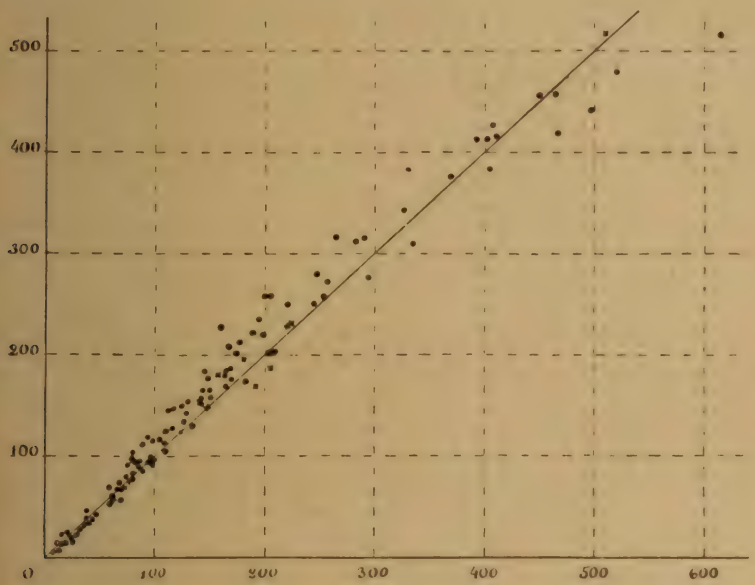


Fig. 2.

operated each time with 20 similar test pieces; the mean of the 20 resistances has been calculated, as well as the difference between each separate resistance and the mean; also the mean of the absolute value of these differences and the ratio between the mean difference and the mean resistance.

The rammed mortars (1 : 2 : 3) were made as described at the beginning of chapter A, immersed after 24 hours and broken by tension at the end of 28 days.

The plastic mortars (50 : 200 : 750) were moulded into prisms measuring $16 \times 4 \times 4$ cm, immersed after 7 days, and broken after 28 days. The bending tests were carried out under constant

moment with the apparatus shown in fig. 1, the distance AA' being 4 cm*). Those by compression were made on a half of each prism, by means of Le Chatelier's registering Dynamometer.

The following table gives the means of the resistances and differences given by the 20 tests of each kind.

Nature of the puzzuolana	Percentage retained by the 4900 sieve	Rammed mortars 1 : 2 : 3				Plastic mortars 50 : 200 : 750							
		Mixing water	Tension			Mixing water	Bending			Compression			
			Resistance	Difference absolute	Difference relative		Resistance	Difference absolute	Difference relative	Resistance	Difference absolute	Difference relative	
			kg/cm²		p. 100		kg/cm²		p. 100	kg/cm²		p. 100	
Quenched slag	15	0,90	22,6	1,22	5,4	138	20,8	0,95	4,6	81,4	4,15	5,1	
Another quenched slag .	25	0,93	18,6	1,22	6,6	140	14,7	0,77	5,2	41,6	2,10	5,0	
„Si-Stoff" (a sifted industrial residue)	50,5	1,26	18,3	1,00	5,5	188	14,1	0,57	4,0	38,3	0,91	2,4	
Roman puzzuolana .	24,5	0,96	13,1	0,63	4,8	135	15,0	0,45	3,0	42,8	2,06	4,8	
Baked gaize	24	1,35	8,9	0,74	8,4	190	7,7	0,26	3,5	15,2	0,29	1,9	
Averages:					6,1				4,0			3,8	

We see that the tests with plastic mortars gave very concordant results, since the mean of the differences was only 4 per cent of the resistances found. The tensile tests of rammed mortars gave an average difference of about 6 per cent, but perhaps the results would have been more concordant if the ramming had been done mechanically. We remark, on the other hand, that the order of classification of the five puzzuolanas is not quite the same for the two kinds of mortar.

The degree of sensitiveness of a testing method may also be judged of by the differences between the results it furnishes when applied to products known to be of different qualities. To the experiments of chapters A and B, already very favourable to the plastic mortars, we add the following, which were executed like the last five except that there were fewer test pieces of each mortar. They show well the differences in resistance that can be got with different puzzuolanas. In particular, tests 7—13, made with specimens of trass specially sent by M. Herfeldt, show clearly the differences observed in practice between the first and the last of them.

*) In the conditions under which the tests with a central load described above, and also those with a constant moment (with which we are here concerned), were carried out, the former generally give rather greater resistances than the latter.

No. of the test	Nature of the puzzuolana	Left on the 4900 sieve	Mixing water gms. per kg	Resistance per cm²	
				bending	Compression
1	Trass from an old source at the port of Calais	34	148	10,3	24,3
2	Trass from another source at the port of Calais	29,5	148	10,5	26,8
3	Trass from an old source at the port of Dunkirk	46	148	9,1	21,1
4	Trass from another source at the port of Dunkirk	50	148	7,5	17,8
5	Same as the last but passed through the 324 sieve	41	148	8,3	19,0
6	Trass of good quality sent ground by Herfeldt in 1896	16	148	13,5	36,9
7	Good trasses in lumps from the Nette Valley — yellow	34,5	148	9,5	25,5
8	Good trasses in lumps from the Nette Valley — grey	33	148	11,3	31,9
9	Good trasses in lumps from the Nette Valley — blue	32	148	12,9	35,4
10	Mixture of the last 3, 1/2 yellow, 1/4 grey, 1/4 blue	33,5	148	11,1	29,4
11	False trasses (Leucite-tuff) from Weibern	28,5	148	4,3	11,0
12	False trasses (Leucite-tuff) from Rieden	31	148	7,1	17,0
13	False trasses (Leucite-tuff) from Bell	30	148	8,6	25,4
14	Roman puzzuolana	24,5	135	15,0	42,8
15	„ „ from another quarry	15,5	132	16,2	41,4
16	Puzzuolana from Bacoli near Naples	21	153	6,1	13,0
17	„ „ the Azores	35,5	160	9,6	23,5
18	Santorin earth	24,5	154	3,3	8,0
19	Puzzuolana from the Haute-Loire	14,5	122	2,4	5,4
20	Raw gaize	33	190	3,7	9,2
21	„ „ (another sample)	29	195	2,5	7,7
22	Baked gaize	24	190	7,7	15,2
23	„ kaolin	(2)?	160	16,9	45,8
24	„ „ (another sample)	(1)?	257	12,1	34,3
25	Ordinary clay baked in powder in a muffle	28,5	190	9,4	20,6
26	Broken brick	24,5	142	2,9	6,9
27	„ „ put through the 4900 sieve	0	150	4,5	8,7
28	Si-stoff, a sifted industrial residue	50,5	188	14,1	28,3
29	Artificial puzzuolana, a kind of quenched slag	27,5	135	25,0	82,3
30	Another puzzuolana, a kind of quenched slag, another composition	8,5	138	16,4	46,2
31	Quenched blast furnace slag, works A	12,5	135	22,1	86,8
32	„ „ „ „ „ A	25	140	14,7	41,6
33	„ „ „ „ „ A	24	140	18,8	56,6
34	„ „ „ „ „ B	20,5	135	23,8	79,5
35	„ „ „ „ „ B	12,5	135	28,0	94,3
36	„ „ „ „ „ B	15	135	23,2	79,5
37	„ „ „ „ „ B	20,5	135	20,2	60,5
38	„ „ „ „ „ C	11,5	135	25,1	96,5
39	„ „ „ „ „ C	25,5	135	23,5	95,3
40	„ „ „ „ „ C	15	138	20,8	81,4
41	„ „ „ „ „ D	12,5	135	19,0	55,3
42	„ „ „ „ „ E	23,5	135	7,2	13,3
43	„ „ „ „ „ F	33	135	17,5	46,8
44	Foundry slag, calcareous in character and cold (works F)	11	130	20,0	60,3
45	Quenched blast furnace slag (F) finery slag	14	140	5,4	9,3

In comparing the numbers in this table attention must be paid to the difference in the grinding of the various samples, which were not all reduced to the standard fineness. Besides, care must be taken not to pronounce too hastily that such or such a puzzuolana is superior to another, for many puzzuolanas are only represented by one or two samples, which may be different from the average quality of the particular kind.

K. Accessory arrangements for resistance Tests.

There is nothing to change in the proposals of the programme, except perhaps the ratio of the number of bending experiments to compression experiments, which will depend on the length of the prism finally adopted.

Besides, it will be advisable to stipulate that the prisms must be immersed in such a position that their opposite faces, which were in contact, during mixing, with the sides of the mould and which are subjected to the maximum tension and compression in the bending test, are vertical. In fact, various experiments have shown us that when these faces are horizontal in the water, the upper one sometimes hardened rather faster than the lower one, so that the breaking load was greater or less according to whether the upper or lower face was subjected to tension.

II. Practical Tests.

L and M. Granulometric composition and homogeneity Tests.

There is nothing to change in the proposals of the programme.

Examples of determination of granulometric composition are given above in chapter A.

Homogeneity tests were described in the appendix to the programme, and in an article headed «Doit-on pulvériser les puzzuolanes?» (Should puzzuolanas be pulverised?)[1]).

N. Resistance Tests.

The same article contains a report on resistance tests made with various mixtures of one lime, one coarse puzzuolana, and one sand. Tests of the same kind have since been made with two puzzuolanas finely powdered, two limes and one sand, mixed three by three in proportions regularly varied.

After preliminary trials the quantities of water were fixed proportionally with the weights of the 3 materials in every mortar, as follows:

With fat lime (Lime A of chapter B) . . . . . 74 per cent of water
„ hydraulic lime from Teil . . . . . 48 „ „ „ „
„ baked gaize leaving 23,5 per cent on the 4900 sieve 49 „ „ „ „
„ trass leaving 22,5 per cent on the 4900 sieve . . 40 „ „ „ „
„ shelly sand in grains 2 mm to $^1/_2$ mm (B′ of chap. C) 6 „ „ „ „

These proportions always gave very plastic mortars which were moulded into prisms of 16 × 4 × 4 cm. These were immersed after 7 days of setting in damp air, and tested, some after 28 days after gauging, some after one year. The prisms were broken by bending under a central load, and their halves were then tested by compression as explained above.

The mean resistances in the two sets of tests of the four sets of mortars are given by figs. 3—18, in which each mortar

[1]) In the «Matériaux de construction», year V, p. 225 (1900) and in the German translation, Baumaterialienkunde, year V, p. 299 and 322, and in the Ton-Industrie-Zeitung, 1900, p. 1747.

Resistance after 4 weeks.

Bending.

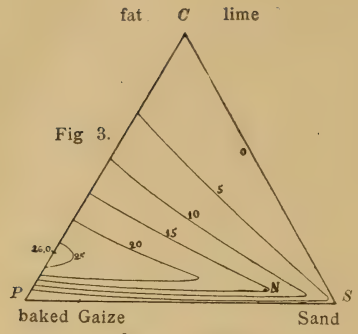


Fig 3.

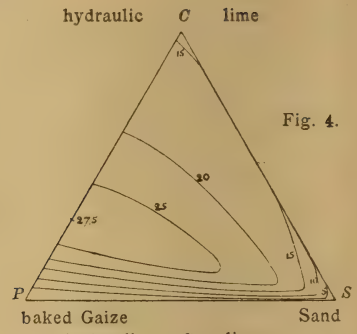


Fig. 4.

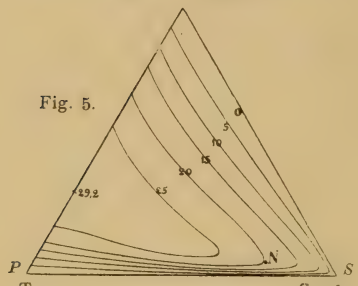


Fig. 5.

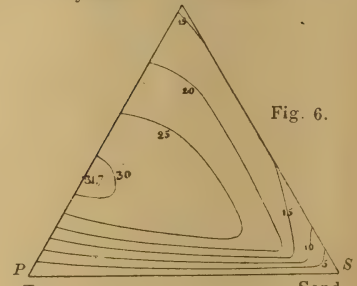


Fig. 6.

Compression.

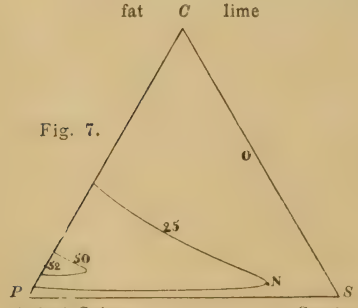


Fig. 7.

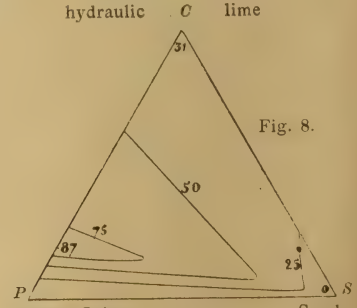


Fig. 8.

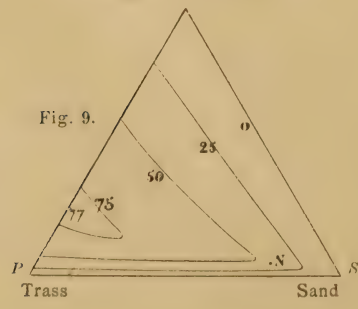


Fig. 9.

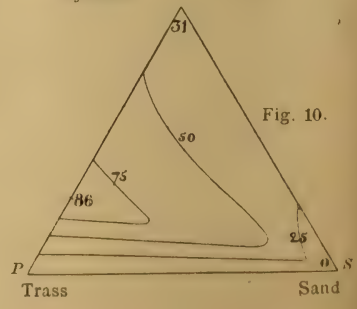
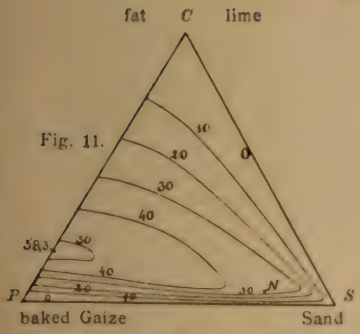
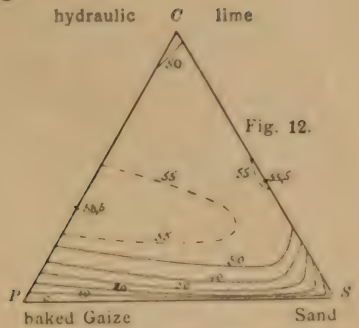


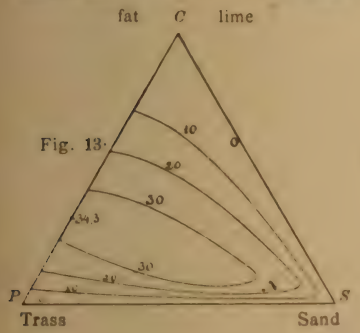
Fig. 10.

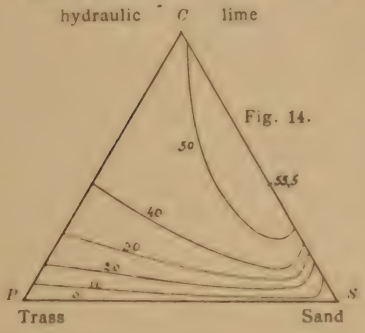
Resistance after 1 year.

Bending.

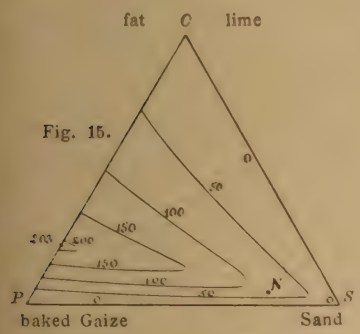


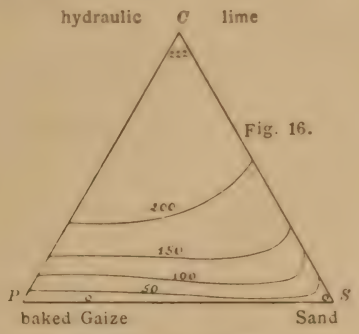


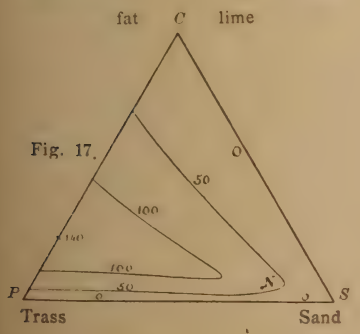


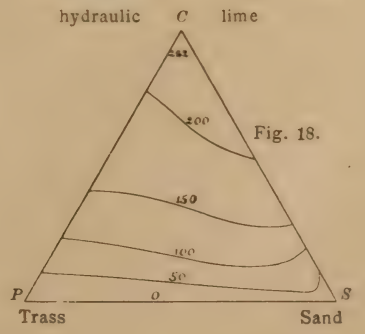


Compression.









is represented by the C. G. of the system of weights got by loading the apices C. P. S. of the triangle with loads proportional to the weights of lime, puzzuolana, and sand, in the mortar.

We observed that, for all the mortars containing the same amount of sand (points on a straight line parallel to CP), the maximum resistance corresponds to proportions of lime and puzzuolana varying with the nature of these materials, and, often, with the age of the mortar For a given lime and puzzuolana these proportions sometimes vary also with the amount of sand. But the maximum resistance always diminishes as the proportion of sand increases.

At the same time, the figures relating to the fat lime mortars give an idea of variations in resistance resulting from choosing standard proportions different from that (1 : 4 : 15) adopted above (point N of the triangles).

Analogous experiments could always be undertaken with the lime, puzzuolana, and sand, used on the actual work, whatever may be their nature and granulometric composition, and will permit of fixing a composition of the mortar from a full knowledge of the circumstances. But it will rarely be necessary to make such a complete set of tests.

If it is only a question of getting great strength, at any cost, it will generally be obtained with a mortar without sand, and it will be enough to make comparative tests of strength with mixtures of lime and puzzuolana in graduated proportions.

If, for economy, or other reason, sand is to be used, but there is no a priori knowledge of how much to put, it will be best to proceed according to our preliminary programme, and to try merely a few mortars, represented in the triangle by apices of a series of smaller triangles. The results obtained may lead to a trial of new mortars, forming the apices of a set of smaller triangles, and these will have compositions approximating to that of the mortar which gave the best results in the first set of tests.

If the quantity of sand to be taken is already known approximately, it will be best to begin by mixing a set of mortars containing for the same weight of sand the same weight of mixtures consisting of various proportions of lime and puzzuolana, and with the proportion giving the highest resistance, to make a new series of tests, varying only the proportion of sand.

O. Setting Tests.

The thumb test proposed above for standard mortars can be applied to any mortar, and is the only possible one to employ in builders' yards. It is necessary to keep as closely as possible to the practical conditions under which the mortars are used, especially as to the place of storage and the temperature of the locality. Experiments made a few years ago in Germany tend to show that the setting of lime-puzzuollana mortars is more affected by changes in temperature than that of cement mortars.

P. Deformation Tests.

There is nothing to modify in the proposals of the programme.

We have given in chapter B the report on the cold and hot dilatation tests made in cylinders fitted with needles, with pastes of puzzuolana and various limes without any sand. None showed any very marked expansion but the case would have been very different if the limes had been badly slaked.

Q. Adhesion Tests.

We shall not here insist at length on the method proposed for these tests, which is described and discussed in a special report. The method consists in moulding the mortar to be tested on to the body, the adhesion to which is to be tested, so as to form a prism, which is tested by bending under a constant moment in the apparatus shown in fig. 1.

For puzzuolana mortars, it is clearly advisable to adopt the same method as for cement mortars.

We have made two sets of tests with puzzuolana mortars, the first to compare the adhesiveness of various mortars to the same body, the second to compare the adhesiveness of the same mortar to various bodies.

In the first series, the adhesion bodies were blocks of mortar about 10 years old, and composed of one part cement passed through the 900 sieve and two parts silicious sand passing the 144 sieve but not the 324 sieve. The mixture was tempered with one tenth of its weight of water and rammed into prismatic moulds of 8 × 4 × 4 cm. The moulded mortar was kept by turns in damp air and soft water. Before using these blocks the surface of

one end was freed from incrustations and made perfectly plane by grinding, and they were soaked for a few hours in water. The prisms were then placed horizontally in moulds of twice their length. The rest of the mould was then filled up with plastic mortars, 50 : 200 : 750, made of the same fat lime and various finely ground puzzuolanas, so that the fresh mortar abutted against the vertical planed end of the adhesion body. The prisms were kept 7 days in damp air, and then 21 days in water, and tried by a constant bending moment in the apparatus shown in fig. 1 All broke cleanly along the joint between the two different substances. Finally the half prisms consisting of puzzuolana mortar were tested by compression between two plates of steel 4 cm wide.

Each time, six prisms were tested, and the results are given in the following table, in such a way as to show differences to be expected. It is unnecessary to give the details of the six compression tests, which agreed very better among themselves.

Designation of the puzzuolanas	Grammes of mixing water per kg of dry mixture	Adhesion tests				Compression tests		
		Resistances in kg per cm²		Mean of the differences		Mean resist. per kg/cm²	Mean of the differences	
		individual values	Mean	absolute	relative		absolute	relative
		kg	kg	kg	p.100	kg	kg	p.100
Trass	148	4,1 4,0 3,3 5,4 4,6 3,7	4,2	0,66	15,7	24,6	0,98	4,0
Roman puzzuolana	135	8,7 7,4 7,4 5,4 4,0 5,4	6,4	1,74	27,2	45,5	1,53	3,4
Another sample . .	140	(See the 2d series of tests)	4,4	0,64	14,5	33,6	2,38	7,1
Bacoli puzzuolana.	153	3,6 3,3 3,5 4,1 3,3 3,4	3,5	0,22	6,3	14,6	0,35	2,4
Raw gaize	190	1,3 2,3 — (1) — —	1,8	0,50	27,8 [1])	10,8	0,22	2,0
Baked gaize	190	3,3 6,2 6,8 3,4 5,5 —	5,1	1,34	26,3	16,1	0,37	2,3
Baked kaolin . . .	160	5,3 4,6 8,0 6,5 — —	6,1	1,15	18,9	61,3	2,83	4,6
Broken brick	142	2,8 2,3 2,1 1,6 3,0 —	2,4	0,44	18,4	7,4	0,23	3,1
Si-Stoff	188	4,0 5,5 5,7 6,5 3,8 5,5	5,2	0,83	16,0	34,4	1,13	3,3
Blast furnace slag quenched	140	5,4 7,7 7,3 7,5 7,9 —	7,2	0,76	10,6	36,6	1,03	2,8
Another sample . .	138	8,0 11,5 9,2 12,1 10,4 9,5	10,1	1,22	12,1	84,7	3,67	4,3
					Average: 16,6		Average: 3,6	

[1]) The remaining four prisms separated in the moulds on account of shrinking of the mortar as it sets. So few results having contributed to the average, it has not been reckoned in the main of all relative differences.

In the second set of tests we used a plastic mortar containing 50 of fat lime, 200 of a Roman puzzuolana ground, as to leave 27 per cent residue on the 4900 sieve, and 750 of sand consisting of equal weights of grains from 2 to $^1/_2$ mm. The water added was 140 parts.

The adhesion bodies were:

1. The same as in the preceding tests, i. e. a Portland cement mortar 1 : 2, 10 years old.

2. A mortar consisting of Portland cement mixed with three times its weight of a rather fine natural sand to a plastic consistency and kept for a year, first in water, then in damp air. The ends were planed as above.

3. Four different kinds of stone cut into prisms of 8 × 4 × 4 cm.

The prisms were moulded and broken by bending as above described. Six half prisms consisting of the puzzuolana part were tested by compression, and gave an average resistance thereto of 33·6 kg per cm^2 with a mean difference between the individual results of 2,38 kg, i. e. 7,1 per cent of the mean resistance.

Nature of the adhesion body	Adhesion tests			
	Resistance per cm^2		Mean difference	
	individual values	Average	absolute	relative
	kg	kg	kg	p. 100
Rammed mortar 1:2 Portland cement, ten years old	5,0 5,1 5,7 3,8 3,4 4,7 4,2 4,3 / 6,1 4,3 4,1 2,9 3,6 4,9 4,1 4,1	4,4	0,64	14,5
Plastic mortar 1 : 3 Portland cement, one year old	5,1 5,3 4,3 5,0 5,1 7,4	5,4	0,70	12,9
White marble	3,3 3,3 3,2 5,4 4,1 —	3,8	0,70	18,4
Marquise limestone	3,6 4,7 3,1 4,1 5,2 3,8	4,1	0,58	14,1
Jeumont stone	2,1 1,7 2,3 2,0 4,1 —	2,4	0,64	26,6
Vergelé	1,6 1,0 1,4 1,5 2,1 —	1,5	0,26	17,3
			Average:	17,3

Hence the adhesion depends both on the nature of the adhesion body and on the composition of the mortar. Moreover the results of adhesion tests never agree so satisfactorily as in the tension, bending, or compression tests.

We explain in the special report that the differenc es observed are not due to any want of precision in the method, but correspond to real differences between the adhesions of the various prisms in the same series, differences which may be more or less marked according to the composition of the mortar.

Instead of operating as in the preceding tests on prisms of a total length of 16 cm., it will be best to give that length to each of the half prisms, so that after breakage we can submit the mortar half to the bending and compression tests. We should then fix at 20 cm the distance between the contacts of the clamps on the compressed face of the whole prism, and should be thus nearly sure that no force would occur at the joint of separation but those resulting from the bending force purposely applied.

When the prisms are made, a well stretched piece of wet paper should be spread over the bottom of the mould, so as to prevent accidental breakage in removing them from the moulds.

Recapitulation of the proposed rules.

The trials of puzzuolanas may be undertaken either to determine the intrinsic value of any particular puzzuolana and to make a comparison between sundry puzzuolanas, or to give information to the builder as to the results he may expect to obtain with the puzzuolana he proposes to use, at the degree of fineness to which he can bring it, with the plant at his disposal.

In judging intrinsic merits, or comparing two or several puzzuolanas, the trials should always be made under identical and therefore perfectly defined conditions; in particular the puzzuolana should be of a well defined and constant degree of fineness. These trials may be called «characteristic» or «standard».

In trying a puzzuolana for the builder's instruction, greater latitude may be given to the experimenter, but, when there is no reason to the contrary, the normal rules should be observed as closely as possible. In all cases the report of the trials should state the methods employed. These trials may be called «practical» trials.

1. Characteristic Trials.

Standard fineness. The puzzuolana to be tried should be brought by the simultaneous pulverisation of all its parts, and without sorting out any of the grains, to such a state that when passed through a sieve with 4900 meshes per cm^2 from 20 to 30 per cent of its weight is retained on the sieve.

For the description of the sieve and the method of using it refer to the directions relative to the trials of cements.

Standard fat lime. Fat lime, thoroughly slaked in powder and sifted, and answering the following conditions must be used:

The solid impurities in the lime must not exceed 3 per cent of the weight of the lime (carbonate of lime excepted), and the carbonate of lime shall not exceed 7 per cent.

The proportion of water contained must be between 24·3 and 30 per cent of the weight of the hydrated lime, calculated after deducting the solid impurities and the carbonate of lime.

The lime must leave no residue on a sieve of 900 meshes per cm^2.

Standard sand. The standard sand must be the same as that used in the trials of cement.

Provisionally, and awaiting the adoption by the International Association, of a standard sand for cements, a silicious sand with round grains composed of equal weights of three sizes of grains determined respectively by colanders with circular holes 2 mm, 1·5 mm, 1 mm and 0·5 mm, in diameter, is to be used for puzzuolanas.

Composition of the mortar. The mortar must be composed of one part by weight of standard fat lime, four parts of puzzuolana, and fifteen parts of standard sand.

Mixing the mortar. For each mixing we commence by weighing out 50 grammes of lime and 200 grammes of puzzuolana; these are thoroughly mixed in a dry state in a capsule, with a spatula. Then 750 grammes of standard sand is added and mixed afresh. The mixture is spread out on a non-absorbant table, it is hollowed out into the form of a crown, the whole of the water judged necessary being poured then at one operation into the middle of the mass. The method of arriving at the necessary proportion is given in the next paragraph. The water is mixed with the solid matter by means of a trowel held in one hand, and the mortar thus obtained is vigorously stirred for a total period of five minutes counting from the time the water was poured in.

Mixing water. A flat bottomed metal box in the form of a truncated cone 8 cm in diameter at the bottom, 9 cm in diameter at the top and 4 cm in depth, is filled with part of the mortar thus obtained. The mortar is thrust into every part of the box with the finger and, when it is quite filled, the surface is smoothed with the trowel.

A clean and dry sounding rod of polished metal, 1 cm in diameter and weighing 2 kg, terminating in a plain and perpendicular section, is allowed to sink steadily into the centre of the mass thus formed, carefully and without letting it acquire any speed. This instrument, known as a consistency sounding rod, should be constructed in such a manner as to indicate exactly the thickness of the mortar remaining between the bottom of the box and the lower end of the rod.

Two trials must never be made on the mortar of the same filling.

When the consistency of the mortar is such that the distance between the bottom of the box and the lower end of the rod at the moment it ceases to sink by its own weight is 6 mm*), the mortar is considered to be standard.

The quantity of water found necessary to obtain this degree of consistency is noted, and this proportion should be used for all other mixtures made with the same materials.

Setting test. The setting test is made with standard mortar as we have just defined it.

At the moment of mixing, the solid materials, the water and the ambient air (surrounding atmosphere) should be at a temperature between 15 and 18 deg. C.

Immediately after it is made, the mortar is introduced and packed into two boxes as just described, and with the same precautions.

As soon as they are filled, the boxes are taken into a place in which the atmosphere is saturated with moisture and sheltered from draughts of air and from the direct rays of the sun. The temperature of this place must be between 15 and 18 deg. C.

By the setting period of the mortar is meant the time that elapses from the beginning of the mixing operation until the moment when, the box being placed upon the table, no visible depression can be made in the mortar when the thumb is pressed upon it with all the strength of the arm, but without jerking.

One of the boxes will serve for the first trials, and the other should be preserved intact for the end of the operation when the thumb shows only a faint imprint on the mortar.

In case of dispute, the setting test should be replaced by a resistance test, and it will be admitted that the final setting of standard mortar, as defined above, corresponds to a resistance to pressure of 5 kilos per cm^2.

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*) Possibly this figure will be modified when the question of a standard sand is definitely settled.

Standard prisms for resistance tests. For the resistance tests, prismatic test-pieces, square in section, are to be used, 4×4 cm wide, and 16 cm long*).

Moulds, the interior of which is in the shape of these prisms, are placed on a marble slab or on polished metal, and both the mould and the slab are first cleaned and wiped with a greasy cloth.

Two moulds at a time are filled with mortar of the same mixing, the mortar being thrust into every part of the interior with the finger and the surface smoothed with the trowel. The mortar is left to harden in the mould in a moist atmosphere, sheltered from draughts and from the direct rays of the sun, at a temperature as regular as possible between 15 and 18 deg. C.

Twenty four hours after the mortar has set it is taken out of the moulds, and left in the same moist atmosphere.

A week after mixing, the prisms are immersed in drinking water, taking care that the sides which were vertical in the moulds are still vertical in the bath. The water must be maintained, as closely as possible, at between 15 and 18 deg. C. and renewed every week.

The breakage trials should be proceeded with four weeks after the mixing and should be repeated if possible after more prolonged periods, 12 weeks, 26 weeks, a year, two years, and so on.

A sufficient supply of similar prisms should be made on the same day to enable four to be broken at the end of each of the predetermined intervals.

They must be first broken by bending, and the pieces must then be tested by compression.

Apparatus for bending tests. The prisms must be broken under constant bending moment, by means of an apparatus similar to that shown above in fig. 1 and embodying the following conditions.

The distance between the two jaws of the lower clamp must be such that the prism is quite vertical when two opposite faces of it are in contact with them. The two clamps must be exactly

*) This dimension is proposed provisionally and subject to change if fresh experiments show any reason for modifying the distance between the lines of bearing on the compressed face of the prism. In this case the weights of materials to be taken for each mixing will also have to be changed so that the volume of mortar obtained will slightly exceed that of one or of two prisms as the case may be.

4 cm thick and will bear on the prism along four straight lines perpendicular to its length. Along these lines the radius of curvature of the parts of the clamp will be at least one cm. The vertical distance between the two lines of contact of the same clamp (*ab* and *a'b'* in fig. 1) must be about 4 cm.

The lever must be so made that, when the prism is vertical and the upper clamp is in contact with two opposite faces of it, the horizontal distance from the point of suspension of the bucket to the vertical axis of the prism is 54 cm.

The lever must be so balanced that, when in the position above indicated, the C. G. of the lever, combined with the suspensory parts of the bucket not weighed therewith, is on the same side as the bucket and 2—3 cm from the vertical axis of the prism.

Rupture by bending. The prism must be held vertically by the clamps, which must grip the faces which were vertical during moulding and soaking.

The vertical distance (AA' in fig. 1) between the lines of contact nearest to the middle of the prism on the compressed face must be 4 cm*).

Lead pellets will be poured into the bucket at the end of the lever at the rate of 100 grammes per second, by an arrangement which will stop the flow on the prism breaking.

By multiplying by 5 the weight of bucket and lead at this moment we get the number expressing the resistance to bending in kg per cm^2.

Compression tests. The pieces of the prisms broken by bending are placed crossways between two plates of steel 4 cm wide and rather more than 4 cm long, exactly over one another, so that the part of the prism bounded by planes tangential to the edges of the plates is a cube of 4 cm side. In this position the faces of the prism which touch the plates must be those which were vertical during the moulding and the soaking.

*) A number proposed provisionally, until fresh experiments permit of a more scientific determination of this distance.

It matters little from the point of view of the results obtained, whether the ends of the prism touch the bottom of the grips entirely or partly, or not at all. The most convenient plan is to recess the grips inside at a distance of 2 cm from the point of contact with the face of the prism, so that the prism need only bear against the bottom of the grips at its ends, to get the desired distance between the lines of contact with the compressed face.

A uniformly increasing pressure will then be exerted perpendicularly to the steel plates.

The increase must be the same as directed for cement-testing. By dividing by 16 the load causing fracture, we get the number expressing the resistance to compression in kg per cm^2.

2. Practical Trials.

Granulometric composition. With a weight of material increasing with the coarseness of the puzzuolana, the total proportion of grains retained by the 4900 sieve, and the proportion retained by the 900 sieve must be determined, and also the proportion passing through regular circular holes 0·5 mm, 2 mm, 5 mm, 10 mm and 20 mm in diameter.

With puzzuolana in fine powder, each sifting must be done according to the rules for cement testing.

Tests of homogeneity. A simple examination with the naked eye, or a magnifying glass, will often show the presence of foreign grains in a puzzuolana.

For a more searching test, the puzzuolana may be separated into grains of different densities by the liquids used for the purpose by mineralogists. The nature of the different sorts can then be determined by analysis.

If it be possible to separate the puzzuolana into several sorts of grains on a large scale, it may be useful either to grind the various products of separation to the standard fineness and to subject them to the characteristic tests, or to subject to parallel practical tests the puzzuolana in its crude state and the same freed from ingredients supposed to be of inferior quality.

Resistance tests. These are to be carried out first by bending then by compression in the same way as in the characteristic tests, with prisms of the same size made in the same way, but of one or more of the actual materials used in the builder's yard: We may also, in special cases, vary the conditions to which the prisms are subjected between making and breaking them.

When it is a question of determining the best proportions of lime and puzzuolana, or of sand, the same materials as are to be used on the particular job must always be taken.

In the case of sandless mortars, various mixtures of lime and puzzuolana must be compared.

If sand is to be used, and if the amount of it required is approximately known beforehand, it must be mixed with constant weights of various mixtures of puzzuolana and lime and the tests will show the proportion of sand required with reference to those two bodies. Then a new set of tests may be made, varying the amount of sand.

If we have no idea of how much sand is wanted, the first set of tests may be instituted, to compare graduated mixtures of all three ingredients such as

Lime	2	2	2	4	4	6
Puzzuolana	8	6	4	6	4	4
Sand	0	2	4	0	2	0

The results will enable the variations to be restricted to those approximating to the composition which gave best results, and a new set of tests will give the final decision.

In these various tests, the amount of water to be used cannot be ascertained with the sounding rod, which only gives comparative results with mortars of the same granulometric composition. It will be determined by trial for the extreme limits of composition so as to give a plastic consistency similar to that of the standard mortar. The amount of water for the other compositions is found by interpolation. In general, very similar consistencies will be got by making up the total amount of water of parts proportional to the separate weights of the three ingredients.

Setting tests. These will be carried out as with standard mortars, but may be applied, like those of resistance, to mortars different from the standards, either in the nature, coarseness, or proportion of the materials.

The conditions under which the mortar sets must also approximate as closely as possible to those prevailing on the job. In particular, for mortars to be used in cold weather, the tests must be made at low temperatures.

Deformation tests. These must be made with the same mortars as the resistance tests. The mortars must be kept under conditions, the influence of which has to be found, and examined from time to time.

The so-called rapid methods sometimes employed for cements are inapplicable to puzzuolana mortars.

Adhesion tests. The substance, to which the adhesion of the mortar is sought, is made into prisms measuring 16×4×4 ı*). One end of the prism is thoroughly smoothed with emery and well soaked in water before applying the mortar.

Over a plate of marble or metal, a sheet of damp paper must be stretched, on which the prism mould will be set up so that one of its ends is stopped by the prepared end of the adhesion body. This end must have its surface vertical.

The interior of the mould will then represent a prism measuring 16×4×4 cm*).

The moulds are then filled with the mortar to be tested, in the same way and with the same precautions as in making prisms for resistance tests, taking special care that the mortar touches every part of the prepared end of the adhesion body.

The mortar must be allowed to set in an atmosphere saturated with moisture. The mould is then carefully removed, and the compound prisms are kept forseven days in the atmosphere saturated with moisture and then immersed in drinking water, just like prisms for resistance tests.

Twenty eight days after mixing, the prisms are tested, and again at longer intervals if desirable.

Each set of tests must be done with at least six prisms.

The breakage will be done with the same apparatus and with the same precautions as the bending tests, with the one difference that the distance A A' between the contacts on the compressed face must be 20 cm**).

If prisms break along the surface joining their two parts, the adhesion in kg per cm^2 is five times the weight in kg of bucket and lead.

If breakage takes place elsewhere, one can only say that the adhesion is greater than five times that weight.

The broken prisms may be used for bending and then for compression tests.

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*) Dimensions proposed provisionally. They may have to be modified when a final choice has been made of the distance between the contacts on the compressed face in the bending tests.

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 29.

DETERMINATION OF THE LITER WEIGHT OF CEMENT. THE STRENGTH OF REAL HYDRAULIC CEMENTS. DETERMINATION OF A STANDARD SAND.

REPORT OF PROF. F. SCHÜLE,
MANAGER OF THE FEDERAL TESTING
INSTITUTE FOR MATERIALS IN ZURICH.
(TRANSLATED BY R. N. LUCAS.)

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Determination of the liter weight of cement. — The Strength of real hydraulic cements. — Determination of a standard sand.

Report of **Prof. F. Schüle,**
Manager of the Federal Testing Institute for Materials in Zurich.
(Translated by R. N. Lucas.)

I^st. Part.

Determination of the liter weight of cement. The strength of real hydraulic cements.

At the meeting of March 1902, the Council of the International Association determined to invite the Testing Laboratories of Charlottenburg, Paris, St.-Petersburg, Stockholm and Zurich, to study the questions contained in Problem 29, and appointed a reporter to put together the results obtained and present them at the next Congress when held.

At the meeting of March 1903 His Excellency Prof. N. Belelubsky was appointed joint reporter on the question of a standard sand.

The three questions which form the subject of Problem 29 are of very varying significance. The first which is concerned with the determination of the weight of a liter of cement, was nearly solved by the sub-committee B. 22 (Messrs. L. Bienfait, E. Candlot, M. Gary) which had proposed on principle the apparatus of the French commission for determining the liter weight in the shaken and unshaken condition. Difference of opinion prevailed on only one point, namely, the relationships of the metal liter measures in use. These vessels according to the French Commission had a

cylindrical shape with a diameter equal to their height. Prof. Gary accordingly proposed a measure of similar form, but broader and shorter, the ratio of diameter to height being 3 : 2. This ratio possessed the following advantages:

The manipulation of the vessel was easier, shaking down the cement took place quicker and the differences in the different weighings were smaller than with other vessels. Finally these ratios would correspond with the provisions of the German law of weights and measures.

By means of a letter from the Chairman, the attention of the above named Testing Institutes was drawn to the question of determining the liter weight with the request to forward results to the Reporter. As no communications on the subject have come into my hands, I believe I can draw the conclusion that this question has been regarded as of secondary importance and has been settled in each country by the existing laws concerning weights and measures.

The difference in the relationships is in addition not very great. The French liter measure is 108·4 mm in height and diameter. The German liter measure is 124 mm in diameter and 83 mm high. In order to determine if the form of the liter measure has much effect on the weighings both separately and on the average, the weighings were made with a cubic liter measure and a cylindrical measure (d : h = 1) and compared. The latter gave 3 grms. more as the average of the weighings than the liter measure of cubic shape. The difference was the arithmetic mean of 10 weighings of a single cement. The maximum deviation amounted to 5 grms. This experiment will suffice to show the effect of shape and the difference obtainable with one and the same measure.

The Swiss weights aud measures regulation made under the Law of July 3rd 1875 relating to weights and measures ordains that in the case of dry measures the ratio must be d : h = 1 when the measures are cylindrical. The limits of alteration of the theoretic diameter amount to 3% so that the diameter may vary from 105—112 mm.

I propose to the Congress for the purpose of determining the liter weight to accept the suggestion of subcommittee B (§ 3a) and adopt the cylindrical measure in which the height and diameter are equal.

The two other questions which are contained in problem 29 have given rise to extensive researches which are treated of in the sequel. As regards the inherent strength of the binding materials certain conclusions may be drawn from the experiments. As regards the normal sand the question is one of such difficulty that it is not possible to present any complete views of it. The report is accordingly in this respect merely to be regarded as a preliminary communication.

Inherent strength of hydraulic binding materials.

The testing of cements and hydraulic lime is carried out differently in different countries, although these binding materials are practically never employed without admixture of sand. The strengths obtained present accordingly comparatively little importance compared with those of mortar. The ordinances in force in Germany and Switzerland do not mention the inherent strength of the cements. The report of the commission of the American Union of Civil Engineers (Engineering News, Jan. 1903) has not included this test among those which are to be recommended. It may therefore be assumed that the testing of the cements will in time be omitted in the countries like France and Italy where it is at present prescribed.

It was not merely the failure to obtain practical results that led to the abandonment of the experiments. In addition the variations in the strength of the bodies subjected to tractive tests were considerably greater than the differences in the test-pieces of standard mortar or concrete that were to be tested. As these tests belonged to the proceedings of the sub-committee C. 22, and the conclusions (at which they had arrived) indicated two methods of making the tests which led to different results, it proved useful to compare these methods so as to obtain information on the subject and enable the advantages and disadvantages of each to be appreciated.

The sub-committee B expresses itself on this subject as follows:

§ 7. Tension Tests.

d) The moulds consisting of bronze or iron for receiving the cement were placed on a marble table or on a table of polished metal, and rubbed over, as also was the table (after having been thoroughly cleaned), with a greasy rag.

Enough grout was then poured in to fill the mould convexly. Pressure was applied with the fingers so as to remove any cavities and the mould tapped several times on the side with the mallet to complete the sinking down and facilitate the escape of air bubbles.

When the cement had begun to harden it was scraped off horizontally with a straight knife blade. Finally the surface was smoothed off with the knife pressing on the edges of the mould.

e) Another process consists of making a mixture of cement with water until of the consistency of moist earth and pressing it into the mould with a spatula. The two processes give different results.

f) For 24 hours, after mixing, the samples to be subjected to tractive test remained on their plates, in an atmosphere saturated with moisture, and protected from air and sunlight, at a temperature of 15—18° C.

After this length of time the contents of the moulds were removed, the moulds being allowed to glide over the plate unscrewed and removed from the test-pieces.

§ 8. Pressure Tests.

c) The pressure test-pieces must remain 24 hours in the mould both for inherent strength and for mortar strength.

Testing for the strength of pure cements was undertaken in the testing institute of Charlottenburg and in the Zurich institute. Prof. M. Gary published in 1903 the results of the tests made under his supervision. A resumé of these experiments will be given first.

Charlottenburg Tests.

3. Qualities of cements were examined, 2 Portland cements A and B and a Portland cement prepared from blast furnace slag c.

The normal tests gave the following results:

Cements:	A	B	C
Specific gravity	3·206	3·193	3·046
Loss on igniting	1·63	2·53	2·90%
Water added for mixing to normal consistency	25	28·2	29·5%
Commencement of hardening .	6 hrs.	3 hrs.	3 hrs. 45 min.
Time of setting	9½ hrs.	8 hrs.	7 hrs.
Residue through 900 mesh sieve	2·0	1·6	0·2%
„ „ 5000 „ „	18	16	12%
Tension strength after 7 days.	20·7	21·5	20·4 kg/cm²
„ „ „ 28 „ .	25·9	26·8	25·5 „
Resistance to pressure of crushing strain 7 days . .	171·2	131·8	187·8 „
Resistance to pressure of crushing strain 28 days . .	244·6	258·7	285·4 „

Pure cement was examined in two series. The first with admixture of water above the normal consistency (it was poured in), the second at the consistency of moist earth. The test samples of this series were tamped in. Each figure given is the arithmetic mean of 10 separate eperiments, the results are as follows:

Cements:	A	B	C
1st Series (poured in):			
Amount of water	29%	32%	36%
	kg/cm²	kg/cm²	kg/cm²
Tension strength after 7 days.	29·2	27·5	18·0
„ „ „ 28 „ .	54·0	35·4	32·4
Resistance to pressure or crushing strain 7 days . .	352·2	243·6	142·7
Resistance to pressure or crushing strain 28 days . .	554·0	350·3	288·5

2nd Series (tamped in):

Amount of water	17%	19%	20%
	kg/cm²	kg/cm²	kg/cm²
Tension strength 7 days . .	63·6	51·9	51·2
„ „ 28 „ . .	63·2	56·7	65·0
Pressure strength resistance to crushing 7 days . . .	515·4	354·0	366·4
Pressure strength resistance to crushing 28 days . . .	695·6	490·2	549·2

With the amount of water assumed to be present the strength of the tamped in test-pieces is much greater than in the case of those that are poured in. The ratio of the one to the other is as follows for the three cements:

	Tension strength		Resistance to crushing	
Cement:	7 days	28 days	7 days	28 days
A	1·6	1·2	1·5	1·3
B	1·9	1·6	1·5	1·4
C	2·8	2·0	2·6	2·4

In order to compare the two methods it is advisable to determine the difference of the actual values obtained from the mean value. Mr. Gary has accordingly collected the separate values into groups differing by 5% in which the strength varied from 75% to 120% of its mean value.

Values of separate strengths in % compared with the mean strength as unity.

	75–80	80–85	85–90	90–95	95–100	100–105	105–110	110–115	115–120%
Tension strength:									
poured in.	2	3	8	18	17	25	10	8	8
tamped in	0	0	5	17	30	22	20	7	0
Resistance to crushing:									
poured in.	0	2	0	3	48	32	13	0	2
tamped in	0	0	3	13	30	32	20	2	0

From this table we may draw the conclusion that the tamped-in test pieces which have been subjected to tractive tests show

less variation in their behaviour than the test pieces which have been prepared by pouring into the moulds.

The specific gravity, after 28 days, is contained in the following table:

	Poured in		Tamped in	
Cement	Tension	Pressure	Tension	Pressure
A	2·289	2·166	2·405	2·268
B	2·201	2·121	2·362	2·220
C	2·055	1·969	2·201	2·107

Zurich Experiments.

The experiments of the Federal Institute for Testing Materials at Zurich were conducted with three series of Portland cement of Swiss origin prepared by the dry process, with Roman cement, and slag cement. These cements are distinguished by letters or numerals. Roman cements 21 and 32 are of Swiss origin, No. 40 comes from Grenoble.

The experiments include experiments with samples in which the material was poured into the moulds corresponding to the method indicated by the sub-committee B in their conclusions. The amount of water was:

1. That required for normal consistence ($n\%$);

2 this amount diminished by 1% ($n-1\%$) according to the usage of our Laboratory. On the other hand these experiments include the determination of tests with samples in which the cement of the consistency of moist earth had been tamped into the moulds. The amount of water required for this is not exactly determined. Parallel experiments were carried out with a constant amount of water of 20%, which is rather strong for Portland cement, and with a smaller amount corresponding to a drier mixture given empirically by the expression $\left(\frac{n+1}{2}\right)\%$.

The moulds employed were octagonal for tension tests and cubes for the pressure tests. The removal from the mould was according to the recommendation of the sub-committee performed

after 24 hours in the mould in moist air in the case of rapidly setting materials. While in the case of the slow-setting materials the two halves of the moulds were separated 2—3 hours after the contents had begun to harden so, as to avoid the formation of contraction-fissures.

It was of interest to ascertain if it would not be advantageous to replace the usual test-pieces by prisms, as successfully proposed by Mr. Feret. To this end the different cements in normal consistence ($n^0/_0$) were poured into moulds made in six parts and 4 cm high and with a base of 4×16 cm. The tension strength was ascertained by the bending test in the Michaeli apparatus which for this purpose was provided with stirrups or jaws so as to allow 10 cm between the points on which the prisms were supported, the strength as ascertained by the ordinary bending test being divided by 2. The tests obtained in this way can be compared with those by direct tension effort. The crushing strength was obtained by crushing each of the prisms between steel plates 4 cm broad. The tests with „poured-in" test-pieces showed a smaller degree of compactness than in the case of test-pieces made of merely moist cement that had been tamped in. The cause is to be found in the larger proportion of water and the occurrence of air bubbles in the case of the „poured in" test-pieces. We have attempted to avoid these air-bubbles by placing the test-pieces immediately after casting under the air-pump. The result was a negative one; under the diminished pressure the air-bubbles expanded and the surface of the test-pieces rose from $^1/_2$ to 1 cm. If sufficient water is used a point can be reached at which the escape of the air-bubbles under the air-pump is possible owing to the increased fluidity of the mass. The amount of water required for this purpose is $8-10^0/_0$ more than the $n^0/_0$ of normal consistency. The strength of such test-pieces with such an amount of water is noticeably less than when they are of normal consistency. It was on this account that experiments in this direction were not further continued.

Six test-pieces were tested at the same time and the mean of the four highest figures taken in the usual way.

The results of the 7 and 28 day tests are placed side by side in the tables.

Table 1 contains the results of the normal tests of some of the cements examined.

Tables 2 and 3 give the mean strengths (tension and crushing strain) of the cements examined in the various manners mentioned.

Tables 4 and 5 give the relative strengths for tension and crushing strain, unity being taken as the strength after 7 or 28 days of test-pieces of normal consistency.

Table 6 gives data concerning the relative closeness of grain of the test-pieces as mean or average after 7 and 28 days; the unit is the closeness of grain of the test-pieces of normal consistency.

The above tables enable the value of the different methods employed for producing test-pieces to be examined comparatively. Further tables for enabling this comparison to be instituted have also been prepared. Each cement undergoes a normal increase in strength with age. Comparison of the mean strength after 7 and 28 days gives an important element of comparison.

Table No. 7 gives the strengths after 28 days those at 7 days being pre-supposed as unity.

The tables above referred to display a far greater irregularity in the «strength-figures» than is the case with tests of mortar or concrete. These irregularities appear more noticeable in the case of tension tests than in the case of crushing tests. This is mostly shown by the fact that the strength after 28 days only shows a small increase in those found after 7 days.

The maximum value of this ratio corresponds in the case of 16 out of 18 cements examined to the test-pieces for tension and crushing tests which had been poured into moulds. The conclusion accordingly appears justified that in tractive tests the poured-in test-pieces show a more normal increase of strength and correspond more to the increase in strength of the test-pieces when tested for pressure tests.

A further element in the comparison of the various methods is given by the examination of the figures obtained for the different strengths compared with the arithmetic mean of the figures of a series. For this purpose the separate strengths have been divided

into groups, the arithmetic mean of the experiments being taken as unity and introduced into tables 8, 9, 10 and 11 in classes varying from 5 to 5%, the separate strengths varying between 70 and 130%. No distinction was made between the figures for 7 and 28 days in order that the relative value of the different methods might appear more plainly. If for the purpose of reviewing these tables the number of test-pieces the individual strengths of which vary from the mean between the limits 90—110% (for tension strength) and 95 and 105% (for crushing strength) is reckoned in per cents, we obtain:

Water %:	Tension test (Difference ± 10%)	Crushing test (Difference ± 5%)
	for poured-in test-pieces:	
n	61 %	78·5%
n—1	60·8 „	70·9 „
(prisms) n	69·4 „	79·9 „
mean:	64·0%	76·7%
	for tamped-in test-pieces:	
20	69·1%	78·9%
$\frac{n+1}{2}$	73·0 „	74·9 „
mean:	71·8%	76·3%

The means here given show the same result for the test-pieces subjected to crushing tests, but as regards the tractive tests a marked superiority is displayed by the tamped-in method of preparing the test-pieces. As was to be expected the prisms gave the best results, their form being well suited to allow contraction without abnormal interior strains.

The methods of preparing the test-pieces can also be compared in regard to the mean strength obtained. If for this purpose we consider only the Portland cements and leave out of consideration cement 31 the tamped-in test pieces of which gave abnormal results, we can place the mean figures for relative strengths together as follows:

Amount of water % Poured in:	Tension test 7 days	Tension test 28 days	Crushing test 7 days	Crushing test 28 days
n	1·00	1·00	1·00	1·00
n—1	1·02	1·04	1·05	1·04
prisms n	1·23	1·32	1·08	1·04
n 10	—	0·67	—	0·57
Tamped in:				
20	1·10	1·15	1·12	1·05
$\frac{n+1}{2}$	1·42	1·37	1·34	1·19

When it is desired to obtain the maximum amount of strength it is to be recommended for tension tests that there should be used

prisms made with cement of normal consistency

the normal test-pieces with $\frac{n+1\%}{2}$ of water

for crushing tests

cubes with $\frac{n+1\%}{2}$ of water.

For the three Roman cements examined the relative figures for strength are as follows:

Amount of water % Poured in:	Tension test 7 days	Tension test 28 days	Crushing test 7 days	Crushing test 28 days
n	1·00	1·00	1·00	1·00
n—1	1·09	1·00	1·03	1·06
prisms n	1·38	1·18	0·93	1·05
Tamped in:				
$\frac{n+1}{2}$	1·72	1·29	1·77	1·64

The conclusions are the same as in the case of Portland cement although the individual figures are different.

Although the tests for strength were only carried out over periods of 7 and 28 days, some cements were tested for strength after one year in water. The results are shown in tables Nos. 12

and 13. The former gives the absolute strength for tension and crushing tests of Portland cement, three Roman cements, and one slag cement, mixed with $n^0/_0$, $n-1^0/_0$, $20^0/_0$, and $\frac{n+1^0/_0}{2}$ of water, prisms made of pure cement with $n^0/_0$ water were also tested. The relative strengths are given, the strengths after 7 and 28 days and 1 year with $n^0/_0$ water being taken as unity.

This table shows that the alterations in strengths after one year according to the method of preparation employed in the experiments are not substantially different after a year from the results obtained after 28 days and that the above conclusions may apply after a year.

In table No. 13 the increase in strength after 28 days and 1 year is indicated as unity compared with the strength after 7 days. The tension strength of the Portland cements often are less after a year than after 28 days. Roman cement and slag cement are as a rule more regular in their behaviour. In each of the methods employed an increase of strength was noted. The crushing tests show little difference between a year and 28 days. The relative strengths of «tamped in» normal mortars 1 : 3 are also included in this table. It is only exceptionally that Portland cement shows the growth in strength of normal mortar after 28 days. The strength as regards crushing strain shows an increase which may even surpass that of normal mortar.

The experiments with Roman cement show that the growth in strength either for tension or crushing tests and either after 28 days or a year is greater than in the case of normal concrete or mortar.

Conclusions.

The experiments in regard to the strength of pure cements which were undertaken in accordance with the methods proposed by sub-committee B 22 prove that the results vary. The tests of pure cement of normal consistency poured into the moulds, show less strength than tests made from test-pieces in which the cement was tamped into moulds moistened to the consistency of moist earth.

The employment of prisms of 4 by 4 by 16 cm poured into the moulds in normal consistency provides advantages for determining the tension strength by the bending test. Dividing the „strength" figures obtained by the usual bending formula by 2 gives the strength as compared with what is obtained by direct tension test. The advantage of the prism consists in the shape permitting alterations of form without giving rise to abnormal internal strains, such as occur in the case of test-pieces of the usual octagonal shape.

The tension test is, as a rule, unsuitable for showing the increase in strength with the age of the test-piece. The differences of the individual test-pieces in strength as compared with the mean strength are very much greater in the case of tension tests than in the case of the crushing tests, they are greater in the case of the test-pieces made by tamping the material into the moulds, with the exception of the prism tests which do not greatly differ from these made in the usual way with tamped-in test-pieces. In spite of the advantages of the tests with tamped-in test-pieces which have just been given, it is advantageous to take into consideration the much greater ease in preparing the test-pieces when the material is mixed with sufficient water to form a „grouting" which can be poured into the moulds. It must also be borne in mind that the greatest increase in strength among the 18 different Portland cements examined, was found in the case of test-pieces in which the material had been poured into the moulds, this being found after 28 days and the same was proved of 16 samples examined after 7 days. It should be further mentioned that in countries where the inherent strength test is prescribed (in tenders) these are made with test-pieces poured into the moulds in normal consistency.

We accordingly propose to the Congress to adopt the following resolution:

The strength test of real cement does not give the same certain results as in mortar tests.

As a rule, in order that the results may be compared with one another it is recommended that the test should be carried out with test-pieces of normal consistency according to the conclusions of sub-committee B 22.

Where it is a matter of attaining specially great strength the method of tamping-in the material in the consistency of moist earth is to be recommended. The amount of water must be somewhat higher than the half employed for normal consistency i. e. $\frac{n+1}{2}\%$.

The employment of prisms of $4 \times 4 \times 16$ cm which were first bent and then tested under pressure gives results comparable with those obtained with tamped-in test-pieces when the numbers obtained by the ordinary bending formula are divided by 2.

Table No. 1.

Results of the normal tests of the cements employed.

No.	Marks No.	Specific gravity	Loss on ignition %	Setting		Fineness	Normal concrete strength 1 : 3 kg/cm²			
				Hardening begins h	completely set h	Residue on 4900 mesh sieve	after 7 days in water		after 28 days in water	
							Traction	Crushing	Traction	Crushing
a) Portland cements.										
1	19	3·13	2·15	5·15	16·—	7·7	24·5	305·1	32·5	431·3
2	20	3·14	1·42	4·45	15·—	13·8	27·6	316·5	34·4	402·0
3	27	3·03	3·10	6·—	15·—	18·7	20·8	257·5	25·7	347·3
4	31	3·01	4·30	—·10	1·25	1·0	20·1	198·3	25·2	278·8
5	331	of the same origin as the cement No. 19					—	—	30·8	347·8
6	325	„ „ „ „ „ „ „				20	—	—	36·9	442·3
7	342	„ „ „ „ „ „ „				27	—	—	22·7	232·5
8	330	„ „ „ „ „ „ „				31	—	—	25·6	268·8
b) Roman cements.										
9	21	2·96	8·63	—·5	—·12	6·6	6·9	77·5	13·3	145·0
10	32	3·10	4·01	—·13	—·19	2·4	14·8	150·3	18·0	229·0
11	40	3·09	3·20	—·5	—·10	29·7	10·1	92·3	11·6	108·1
c) Slag cements.										
12	22	2·71	6·70	—·25	8·—	9·9	17·3	134·5	21·5	180·3

Table No. 2.

Mean tenion strengths in kg/cm².

No. of bind. material	Amount of water for normal consist.	Poured in — Amount of water n% after 7 days	n% after 28 days	n—1% after 7 days	n—1% after 28 days	Prisms n% after 7 days	Prisms n% after 28 days	Tamped in — Amount of water 20% after 7 days	20% after 28 days	n+1 after 7 days	n+1 after 28 days	Remarks
a) Portland cements												
19	25	47·3	49·1	40·7	46·0	47·5	51·4	49·1	54·5	49·2	67·5	
20	25	44·5	45·4	49·1	52·4	57·5	43·6	48·1	50·7	40·3	54·9	
27	25	39·8	44·8	32·3	30·9	43·2	54,1	36·8	43·5	50·7	53·8	
31	28	21·6	31·3	21·6	26·3	28·5	27·5	23·0	29·2	20·0	28·2	after 7 d. 28 d
331	24	38·8	37·0	44·3	46·3	44·9	60·5	37·2	43·5	59·8	62·0	17% 44·9 52·0
325	27	40·8	40·6	40·5	46·6	58·1	78·6	47·6	52·6	42·3	51·8	20½ 47·6 47·6
342	23·5	36·8	40·9	37·5	46·2	49·5	51·2	36·5	44·2	55·1	56·2	16 35·4 47·4
330	30	20·4	25·5	20·8	25·4	26·9	31·5	31·3	32·8	30·2	35·9	24 25·9 28·7
255	27	22·0	31·3	24·2	28·7	—	—	—	—	31·0	40·8	n+10% — 23·7
262	27	24·6	33·5	21·8	31·3	—	—	—	—	29·0	38·5	„ — 16·8
267	26·5	19·7	28·0	24·8	35·9	—	—	—	—	36·2	43·8	„ — 20·8
A	28	29·5	40·9	—	—	—	—	—	—	44·1	59·4	
B	28·5	25·4	39·4	—	—	—	—	—	—	48·8	58·7	
C	24	30·7	48·3	—	—	—	—	—	—	55·4	65·6	
b) Roman cements.												
21	48	5·8	11·0	6·6	10·7	8·0	13·0	—	—	12·9	15·8	
32	37·5	13·3	19·2	14·8	20·6	19·5	20·4	—	—	20·1	23·1	
40	31	16·2	19·0	16·9	19·5	21·0	22·4	—	—	23·2	23·3	
c) Slag cements												
22	33·7	17·3	21·8	15·9	21·6	17·6	30·7	—	—	23·0	32·1	

Table No. 3.

Mean crushing strain strengths in kg/cm².

No. of bind. material	Amount of water for normal consist.	Poured in						Tamped in				Remarks	
		Amount of water						Amount of water					
		n%		n−1%		Prisms n%		20%		n+1%			
		after 7 days	after 28 days	after 7 days	after 28 days	after 7 days	after 28 days	after 7 days	after 28 days	after 7 days	after 28 days		
a) Portland cements.													
19	25	608	835	572	853	619	801	630	882	561	778		
20	25	532	688	542	734	603	740	587	722	502	625		
27	25	421	577	443	575	439	565	491	633	512	624		
31	28	285	409	257	404	263	400	227	363	160	279		
331	24	548	753	564	720	568	715	565	741	727	844		
325	27	476	624	493	615	563	701	475	584	522	628		
342	23·5	350	514	371	503	376	526	424	550	523	656		
330	30	236	342	255	349	259	385	307	400	313	440	after 28 days	
255	27	250	345	266	368	—	—	—	—	358	478	n+10%	203
262	27	267	374	306	419	—	—	—	—	413	510	„	216
267	26·5	249	350	280	398	—	—	—	—	429	534	„	192
A	28	345	513	—	—	—	—	—	—	554	706		
B	28·5	293	460	—	—	—	—	—	—	474	602		
C	24	551	862	—	—	—	—	—	—	613	793		
b) Roman cements													
21	48	64	126	68	132	64	134	—	—	175	260		
32	37·5	192	329	195	324	171	342	—	—	250	398		
40	31	210	216	214	246	191	226	—	—	264	357		
c) Slag cements.													
22	33·7	132	189	141	229	150	214	—	—	195	260		

Table No. 4.

Relative strengths by traction.

No. of binding material	Amount of water for normal consistency n %	Poured in — Amount of water n% — after 7 days	Poured in — Amount of water n% — after 28 days	Poured in — Amount of water n−1% — after 7 days	Poured in — Amount of water n−1% — after 28 days	Poured in — Amount of water Prisms n−% — after 7 days	Poured in — Amount of water Prisms n−% — after 28 days	Tamped in — Amount of water 20% — after 7 days	Tamped in — Amount of water 20% — after 28 days	Tamped in — Amount of water $\frac{n+1}{2}$ % — after 7 days	Tamped in — Amount of water $\frac{n+1}{2}$ % — after 28 days	Remark
		unity =										
a) Portland cements.												
19	25	47·3	49·1	0·86	0·94	1·00	1·05	1·04	1·11	1·04	1·38	
20	25	44·5	45·4	1·10	1·15	1·29	0·96	1·08	1·12	0·91	1·21	
27	25	39·8	44·8	0·81	0·69	1·08	1·21	0·93	0·97	1·28	1·20	
31	28	21·6	31·3	1·00	0·84	1·32	0·88	1·06	0·93	0·92	0·90	
331	24	38·8	37·0	1·14	1·25	1·16	1·63	0·96	1·17	1·54	1·67	
325	27	40·8	40·6	0·99	1·15	1·42	1·94	1·16	1·30	1·04	1·28	
342	23·5	36·8	40·9	1·02	1·13	1·34	1·25	0·99	1·08	1·49	1·37	
330	30	20·4	25·5	1·02	1·00	1·32	1·24	1·53	1·29	1·48	1·41	after 28 d
255	27	22·0	31·3	1·10	0·91	—	—	—	—	1·41	1·30	n+10%
262	27	24·6	33·5	0·89	0·94	—	—	—	—	1·18	1·15	„
267	26·5	19·7	28·0	1·26	1·28	—	—	—	—	1·84	1·57	„
A	28	29·5	40·9	—	—	—	—	—	—	1·50	1·45	
B	28·5	25·4	39·4	—	—	—	—	—	—	1·92	1·49	
C	24	30·7	48·3	—	—	—	—	—	—	1·80	1·35	
b) Roman cements.												
21	48	5·8	11·0	1·14	0·97	1·38	1·18	—	—	2·22	1·44	
32	37·5	13·3	19·2	1·11	1·02	1·47	1·17	—	—	1·51	1·22	
40	31	16·2	19·0	1·04	1·03	1·30	1·18	—	—	1·44	1·23	
c) Slag cements.												
22	33·7	17·3	21·8	0·92	0·99	1·02	1·41	—	—	1·33	1·47	

able No. 5.

Relative crushing strengths.

Amount of water for normal consistency n %	Poured in — Amount of water n%		n−1%		Prisms n%		Tamped in — Amount of water 20%		$\frac{n+1}{2}$ %		Remarks
	after 7 days	after 28 days	after 7 days	after 28 days	after 7 days	after 28 days	after 7 days	after 28 days	after 7 days	after 28 days	
	unity =										
a) Portland cements.											
25	608	835	0·94	1·02	1·02	0·96	1·05	1·06	0·92	0·93	
25	532	688	1·02	1·07	1·13	1·08	1·10	1·05	0·94	0·91	
25	421	577	1·05	1·00	1·04	0·98	1·17	1·10	1·22	1·08	
28	285	409	0·90	0·99	0·92	0·98	0·80	0.89	0·56	0·68	
24	548	753	1·03	0·96	1·04	0·95	1·03	0·98	1·33	1·12	
27	476	624	1·04	0·99	1·19	1·12	1·00	0·94	1·10	1·01	
23·5	350	514	1·06	0·98	1·07	1·02	1·21	1·07	1·49	1·27	
30	236	342	1·08	1·02	1·10	1·12	1·30	1·17	1·33	1·28	
27	250	345	1·06	1·07	—	—	—	—	1·44	1·39	after 28 days n + 10% 0·59
27	267	374	1·15	1·12	—	—	—	—	1·55	1·36	„ 0·58
26·5	249	350	1·12	1·14	—	—	—	—	1·72	1·53	„ 0·55
28	345	—	—	—	—	—	—	—	1·61	1·37	
28·5	293	—	—	—	—	—	—	—	1·61	1·31	
24	551	—	—	—	—	—	—	—	1·11	0·92	
b) Roman cements.											
48	64	126	1·06	1·05	1·00	1·06	—	—	2·74	2·06	
37·5	192	329	1·02	0·99	0·89	1·04	—	—	1·30	1·21	
31	210	216	1·02	1·14	0·91	1·04	—	—	1·26	1·65	
c) Slag cements.											
33·7	132	189	1·07	1·21	1·14	1·14	—	—	1·48	1·38	

Table No. 6.

Relative density of test-pieces.

No. of the binding material	Amount of water n%	Traction					Crushing				Remarks
		Poured in			Tamped in		Poured in		Tamped in		
		n% = unity	n − 1%	Prisms n%	20%	$\frac{n+1}{2}$%	n% = unity	n − 1%	20%	$\frac{n+1}{2}$%	
a) Portland cements.											
19	25	2·25	1·02	1·00	1·01	1·06	2·28	1·00	1·00	1·06	
20	25	2·25	1·00	0·99	1·00	1·05	2·23	1·01	1·00	1·07	
27	25	2·21	1·02	1·00	1·01	1·06	2·23	1·00	1·00	1·07	
31	28	2·12	0·99	1·00	1·02	1·05	2·13	1·01	1·07	1·07	
331	24	2·26	1·02	—	1·01	1·08	2·30	1·00	1·02	1·07	
325	27	2·19	1·02	—	1·04	1·08	2·20	1·00	1·05	1·09	
342	23·5	2·32	0·99	—	1·01	1·05	2·30	1·00	1·02	1·07	n + 10% Traction Crush
330	30	2·08	1·02	—	1·06	1·09	2·08	1·01	1·08	1·13	
255	27	2·17	1·01	—	—	1·06	2·16	1·00	—	1·07	0·97 0·9
262	27	2·16	1·01	—	—	1·06	2·15	1·00	—	1·09	0·91 0·9
267	26·5	2·15	1·05	—	—	1·07	2·16	1·00	—	1·09	0·95 0·9
A	28	2·20	—	—	—	1·11	2·17	—	—	1·12	
B	28·5	2·17	—	—	—	1·10	2·17	—	—	1·11	
C	24	2·32	—	—	—	1·04	2·33	—	—	1·04	
b) Roman cements.											
21	48	1·83	1·00	1·00	—	1·08	1·86	1·00	—	1·08	
32	37·5	2·00	1·01	0·99	—	1·09	2·02	1·00	—	1·10	
40	31	2·11	1·00	0·98	—	1·07	2·12	1·01	—	1·08	
c) Slag cements.											
22	33·7	1·92	1·00	0·99	—	1·04	1·93	1·00	—	1·05	

Table No. 7.

Rates of the strengths after 7 and 28 days.

No. of the bind. material	Amount of water	Traction					Crushing				
		Poured in			Tamped in		Poured in			Tamped in	
		n%	n—1%	Prisms n%	20%	$\frac{n+1}{2}$%	n%	n—1%	Prisms n%	20%	$\frac{n+1}{2}$%
		a) Portland cements.									
19	25	1·04	1·13	1·08	1·11	1·37	1·37	1·49	1·29	1·40	1·39
20	25	1·02	1·07	0·76	1·05	1·36	1·92	1·35	1·23	1·23	1·24
27	25	1·13	0·96	1·25	1·18	1·06	1·37	1·30	1·29	1·29	1·22
31	28	1·45	1·24	0·97	1·27	1·41	1·43	1·57	1·52	1·60	1·74
331	24	0·95	1·05	1·35	1·17	1·04	1·37	1·28	1·26	1·31	1·16
325	27	1·00	1·15	1·35	1·11	1·22	1·31	1·25	1·25	1·23	1·20
342	23·5	1·11	1·23	1·03	1·21	1·02	1·47	1·36	1·40	1·30	1·25
330	30	1·25	1·22	1·17	1·05	1·19	1·45	1·37	1·49	1·30	1·40
255	27	1·44	1·18	—	—	1·32	1·38	1·38	—	—	1·34
262	27	1·36	1·44	—	—	1·33	1·40	1·37	—	—	1·23
267	26·5	1·42	1·45	—	—	1·21	1·41	1·42	—	—	1·24
A	28	1·39	—	—	—	1·12	1·49	—	—	—	1·27
B	28·5	1·53	—	—	—	1·31	1·57	—	—	—	1·27
C	24	1·57	—	—	—	1·18	1·56	—	—	—	1·29
		b) Roman cements.									
21	48	1·90	1·62	1·62	—	1·22	1·98	1·94	2·10	—	1·48
32	37·5	1·44	1·39	1·05	—	1·15	1·71	1·66	2·00	—	1·59
40	31	1·18	1·16	1·06	—	1·00	1·13	1·15	1·18	—	1·35
		c) Slag cement.									
22	33·7	1·26	1·36	1·75	—	1·40	1·44	1·62	1·43	—	1·33

Table No. 8.

Tension test.

Frequency of the variations between the individual and mean strengths in %

1. Tamped in.

Total No. of tests	Description of binding materials and ratio of amount of water	70 to 75	75 to 80	80 to 85	85 to 90	90 to 95	95 to 100	100 to 105	105 to 110	110 to 115	115 to 120	120 to 125	12 to 13
	n %												
48	No. 19, 20, 27, 31	2·1	6·3	2·1	16·7	8·3	18·8	14·6	8·3	6·3	6·3	6·2	4·
48	331, 325, 342, 330	4·2	4·3	2·1	4·3	19·2	19·2	12·8	10·6	10·6	6·4	2·1	4·
36	A, B, C	—	5·5	2·8	8·3	22·2	16·7	11·1	16·7	11·1	2·8	—	2·
48	No. 22, 40, 32, 21	—	—	2·1	18·8	10·4	12·5	27·1	16·7	10·4	2·1	—	—
180		1·6	3·9	2·2	12·3	14·5	16·8	16·8	12·9	9·5	4·5	2·2	2
	n — 1%												
48	No. 19, 20, 27, 31	6·3	8·3	2·1	6·3	16·6	16·6	8·3	12·5	8·3	6·3	2·1	6
48	331, 325, 342, 330	4·1	2·1	—	14·6	18·8	10·4	20·9	10·4	6·3	8·3	2·1	2
48	No. 22, 40, 32, 21	—	—	4·2	12·8	17·0	17·0	23·4	10·6	12·8	2·1	--	—
144		3·5	3·5	2·1	11·2	17·4	14·7	17·5	11·2	9·1	5·6	1·4	2
	Prisms n %												
48	No. 19, 20, 27, 31	—	2·1	10·4	12·5	10·4	14·6	18·8	16·7	6·2	6·3	—	2
48	331, 325, 342, 330	2·1	—	—	4·1	25·0	25·0	20·8	4·2	14·6	4·2	—	—
48	No. 22, 40, 32, 21	—	—	—	12·5	22·9	16·7	22·9	10·4	12·5	—	2·1	—
144		0·7	0·7	3·5	9·7	19·4	18·8	20·8	10·4	11·1	3·5	0·7	0
468	General mean:	1·9	2·5	2·6	11·2	17·1	16·9	18·3	11·7	9·8	4·5	1·3	

Table No. 9.

Tension test.

Frequency of variations between individual and mean strengths in %.

2. Tamped in.

Total No. of tests	Description of binding materials and ratio of amount of water	70 to 75	75 to 80	80 to 85	85 to 90	90 to 95	95 to 100	100 to 105	105 to 110	110 to 115	115 to 120	120 to 125	125 to 130
	$\frac{20\%}{}$												
48	No. 19, 20, 27, 31	—	—	2·2	17·4	15·2	23·9	15·2	13·0	6·5	6·5	—	—
48	331, 325, 342, 330	2·1	—	2·1	8·3	18·8	22·9	16·7	12·5	16·7	—	—	—
96		1·0	—	2·1	12·9	17·0	23·4	16·0	12·7	11·6	3·2	—	—
	$\frac{n + 1\%}{2}$												
48	No. 19, 20, 27, 31	—	2·4	2·4	9·5	9·5	21·5	28·6	19·0	4·8	—	—	2·3
48	331, 325, 342, 330	2·1	—	6·4	6·4	14·9	19·2	21·3	12·8	10·6	2·1	2·1	2·1
36	A, B, C	—	—	—	22·2	11·1	22·2	19·4	11·1	11·1	—	—	2·8
48	22, 40, 32, 21	4·2	—	—	6·2	8·3	41·7	25·0	4·2	2·1	6·2	—	2·1
180		1·7	0·6	2·3	10·4	10·9	26·4	23·9	11·8	6·9	2·2	0·6	2·3
276	General mean:	1·5	0·4	2·2	11·3	13·1	25·4	21·2	12·1	8·5	2·5	0·4	1·4

Table No. 10.

Crushing test.

Frequency of variations between individual and mean strengths in %.

I. Poured in.

No. of test	Description of binding materials Ratio of amount of water	80 to 85	85 to 90	90 to 95	95 to 100	100 to 105	105 to 110	110 to 115	115 to 120
	n %								
48	No. 19, 20, 27, 31	—	2·1	16·7	29·2	43·8	8·3	—	—
48	„ 331, 325, 342, 330	—	—	6·2	56·3	29·2	6·2	2·1	—
36	„ A, B, C	—	2·8	8·3	47·2	25·0	16·7	—	—
48	„ 22, 40, 32, 21	—	—	10·4	43·8	37·5	8·3	—	—
180		—	1·1	10·5	44·0	34·5	9·4	0·5	—
	n — 1%								
48	No. 19, 20, 27, 31	—	8·3	10·4	33·4	33·4	14 6	—	—
48	„ 331, 325, 342, 330	—	—	16·6	41·7	29·2	10·4	—	2·1
48	„ 22, 40, 32, 21	—	—	14·6	39·6	35·4	10·4	—	—
144		—	2·8	13·8	38·2	32·7	11·8	—	0·7
	Prisms n%								
48	No. 19, 20, 27, 31	—	4·2	6·2	50·0	29·2	8·3	2·1	—
48	„ 331, 325, 342, 330	—	—	12·5	41·7	39·6	6·2	—	—
48	„ 22, 40, 32, 21	—	6·2	6·3	37·5	41·7	8·3	—	—
144		—	3·5	8·3	43·1	36·8	7·6	0·7	—
468	General mean:	—	2·3	10·8	42·0	34·7	9·5	0·4	0·2

Table No. 11.

Crushing test.

Frequency of variations between individual and mean strengths in %.

2. Tamped in.

No. of test	Description of binding materials / Ratio of amount of water	80 to 85	85 to 90	90 to 95	95 to 100	100 to 105	105 to 110	110 to 115	115 to 120
	20%								
48	No. 19, 20, 27, 31	—	—	6·5	50·0	34·8	8·7	—	—
48	„ 331, 325, 342, 330	—	2·1	12·5	39·6	33·3	12·5	—	—
96		—	1·0	9·5	44·8	34·1	10·6	—	—
	$\frac{n+1}{2}$ %								
48	No. 19, 20, 27, 31	—	5·0	17·5	32·5	30·0	10·0	2·5	2·5
48	„ 331, 325, 342, 330	—	4·3	12·5	31·2	45·8	6·2	—	—
36	„ A, B, C	—	5·5	2·8	38·8	44·5	5·5	2·8	—
48	„ 22, 40, 32, 21		—	14·9	31·9	46·7	2·1	4·3	—
180		—	3·6	12·6	33·3	41·6	6·0	2·4	0·6
276	General mean:	—	2·7	11·5	37·3	39·0	7·6	1·6	0·4

Table No. 12.

Comparative resistance of the test-pieces following the methods of manufacture employed.

No. of binding materials	Amount of water for normal consistency n %	Poured in									Tamped in					
		Amount of water									Amount of water					
		n %			n — 1%			Prisms n %			20 %			$\frac{n+1}{2}$ %		
		7 days	28 days	1 year	7 days	28 days	1 year	7 days	28 days	1 year	7 days	28 days	1 year	7 days	28 days	1 year
		1·00 =	1·00 =	1·00 =												
Comparative tension strengths.																
a) Portland cements.																
19	25	47·3	49·1	51·1	0·86	0·94	0·98	1·00	1·05	1·02	*1·04*	1·11	1·03	*1·04*	**1·37**	*1·22*
20	25	44·5	45·4	44·4	1·10	1·15	1·13	*1·29*	0·96	1·21	1·08	1·12	*1·28*	0·91	*1·21*	1·02
27	25	39·8	44·8	40·1	0·81	0.69	0·61	1·09	*1·21*	1·16	0·92	0·95	1·19	*1·27*	1·20	**1·37**
31	28	21·6	*31·3*	27·4	1·00	0·84	1·04	*1·32*	0·87	0·82	1·06	0·93	1·23	0·92	0·90	1·15
b) Roman cements.																
21	48	5·8	11·0	23·5	1·14	0·97	0·84	1·40	*1·48*	1·09	—	—	—	**2·22**	1·43	*1·28*
32	37·5	13·3	19·2	23·7	1·11	1·07	1·34	1·47	1·06	1·11	—	—	—	**1·49**	*1·20*	*1·21*
40	31	16·2	19·0	29·7	1·04	1·03	1·13	1·30	1·18	1·13	—	—	—	*1·43*	*1·22*	*1·19*
c) Slag cements.																
22	33·7	17·3	21·8	25·2	0·92	1·00	0·96	1·02	1·41	*1·31*	—	—	—	*1·33*	*1·47*	*1·31*
Comparative compressive strengths.																
a) Portland cements.																
19	25	609	836	*1281*	0·94	1·02	0·95	1·02	0·96	0·91	*1·03*	*1·06*	0·95	0·92	0·93	*1·00*
20	25	532	688	1106	1·00	1·07	*1·02*	*1·13*	*1·08*	0·84	1·10	1·05	0·95	0·94	0·91	0·99
27	25	422	577	984	1·05	1·00	*1·06*	1·04	0·98	0·89	**1·16**	*1·10*	0·98	1·22	1·08	1·03
31	28	*286*	*409*	*893*	0·90	0·99	0·89	0·92	0·98	0·86	0·79	0·89	0·88	0·56	0·68	0·96
b) Roman cements.																
21	48	64	126	365	*1·06*	1·05	*1·03*	0·99	*1·06*	0·95	—	—	—	0·99	1·06	0·95
32	37·5	192	329	*761*	*1·02*	0·99	0·99	0·89	1·04	0·90	—	—	—	0·89	*1·04*	0·90
40	31	210	216	719	*1·02*	**1·14**	*1·10*	0·91	1·05	0·96	—	—	—	0·91	1·05	0·96
c) Slag cements.																
22	33·7	131	189	324	1·07	**1·21**	1·08	*1·14*	1·13	*1·12*	—	—	—	*1·14*	1·13	*1·12*

Relative strengths after 28 days and 1 year (the 7 days strengths being taken as unity).

No. of binding material	Amount of water for normal consistency	Traction								Crushing strain								Normal mortar tamped in 1:3			
		Poured in						Tamped in								Tamped in				Traction		Crushing	
		n %		n − 1%		prisms n %		20%		n + 1/2 %		n %		n − 1%		prisms n %		20%		n + 1/2 %					
	n %	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year	28 days	1 year
a) Portland cements.																									
19	25	1·04	1·08	1·13	1·22	1·08	1·09	1·11	1·08	*1·37*	*1·27*	1·37	2·10	*1·49*	2·13	1·29	1·88	1·40	1·92	1·39	*2·28*	1·32	1·72	1·41	2·08
20	25	1·02	1·00	1·07	1·03	0·76	0·94	1·06	*1·19*	*1·36*	1·13	1·29	2·08	*1·35*	2·08	1·23	1·54	1·23	1·80	1·25	*2·18*	1·25	1·52	1·27	1·65
27	25	1·13	1·00	0·96	0·76	*1·25*	1·07	1·18	1·29	1·06	*1·08*	*1·37*	2·34	1·30	*2·37*	1·29	2·01	1·29	1·96	1·22	1·99	1·23	1·78	1·35	2·06
31	28	*1·45*	1·27	1·22	1·32	0·96	0·79	1·27	1·46	1·41	*1·58*	1·43	3·13	1·47	2·90	1·52	2·90	1·60	3·47	*1·74*	*5·37*	1·25	2·24	1·41	2·90
b) Roman cements.																									
21	48	*1·90*	*4·06*	1·62	2·98	1·62	3·16	—	—	1·22	2·32	*1·96*	*5·65*	1·93	5·47	2·11	5·45	—	—	1·49	3·10	1·93	3·96	1·87	4·92
32	37·5	*1·44*	1·78	1·39	*2·15*	1·05	1·35	—	—	1·15	1·42	1·72	3·96	1·66	3·82	*2·00*	*4·00*	—	—	1·60	3·52	1·22	2·41	1·52	3·70
40	31	*1·17*	1·83	1·15	*1·99*	1·07	1·60	—	—	1·00	1·52	1·03	3·42	1·15	3·68	1·18	3·60	—	—	*1·35*	*3·90*	1·14	2·91	1·17	3·99
c) Slag cements.																									
22	33·7	1·26	1·46	1·35	1·53	*1·74*	*1·90*	—	—	1·39	1·43	1·44	*2·46*	*1·61*	2·46	1·43	2·42	—	—	1·33	2·28	1·24	1·77	1·34	1·95

IInd Part.

Determination of a uniform standard sand.

The question of a normal uniform sand was made the subject of a circular letter of the 28th October 1902 by the President of the Union which letter was sent to the Testing Institutions in Charlottenburg, Paris, St. Petersburg, Stockholm and Zurich, in which the method of research to be adopted in accordance with the proposals of Messieurs Martens, Belelubsky and Greil was as follows:

„Attempts shall be made to discover a sand which may be employed as a uniform international basis of comparison in experiments in regard to binding materials".

For this purpose the Committee takes the liberty of addressing to you the question if it is possible to obtain in your country pure quartz sand in sufficient quantity for the requirements of the country, and if a normal sand can be prepared from it.

The international normal sand to be aimed at should be as far as possible, round grained and not artificially reduced in size. It should contain 96% of silica and should not contain any constituents that can be washed out. Such sands should be put through four perforated sheet iron sieves.

a) of 2·0 mm diameter of holes
b) „ 1·5 „ „ „ „
c) „ 1·0 „ „ „ „
d) „ 0·5 „ „ „ „

The residues on the sieves b, c and d are to be mixed in equal proportions by weight and in this way normal sand of three different sizes of grain prepared, while a similar sand containing two sizes of grain only is to be prepared from the residues of sieves b and d.

The Committee begs that the attempts of the Union to carry out its objects may receive support and where possible an exchange of different sands procured in different countries, should be instituted in order to enable a practical opinion to be formed as to whether it will be advisable in future to use only one normal sand or whether it will be possible to make the required sands locally in accordance with the requirements of the international normal sand.

The various Testing Institutes that have been invited to undertake the tests declared their willingness to enter upon such an arrangement and to make experiments in the direction indicated. We give a resumé herewith of the replies which the President of the Union has received.

The Testing Institute in Charlottenburg writes in regard to this subject under date Nov. 26th 1902 as follows:

„The tertiary quartz strata from which the normal sand used in Germany has been prepared are suitable for constituting an international normal sand. The amount of the sand obtainable would be increased by introducing quartz sand of mixed grain. Further sources of sand having been discovered at Freienwalde by boring, no difficulties need be anticipated.

The sand from Freienwalde may be distinguished from all previously known sand by its great purity. After washing it contains 99% of silica, only a small proportion of matter that can be washed away and in general the grains are round. Further particulars in regard to this sand are obtained from the President of the section Gary in „communications" from the royal technical Testing Institutions, year 1896, p. 103 and 1898, p 121.

The Institute intends to sort out two sizes of grain from the Freienwalde rough sand from the residue on sieves b and d and to employ them for a variety of researches. A number of comparative experiments should be made employing sieve c, but in the light of present experience the Institute regards this sieve as dispensable. On the other hand it is to be wished that corresponding researches, as in the „communications" in the year 1898 should be undertaken by other parties. It would probably then become apparent in all probability that, when the constitution of

an international normal sand has been decided upon, little would be gained, as small variations in composition and in the form of the grains have important effect upon the mortar made therewith."

The Testing Institution of Stockholm sent the following communication under date Nov. 4th 1902:

„Hitherto we have in Sweden only had one place at which a pure round grained quartz sand was known, viz., at Axeltorp in Schonen. Perhaps, however, the Royal Geological Survey Office can indicate other spots. Our Swedish cement works as a rule, employ feldspathic sea sand as normal sand and better results have been obtained in the way of strength with this sand than with the German normal sand employed by other testing Institutes

We propose first of all, to give some preliminary experiments with this and other kinds of Swedish sand which have been proposed, and afterwards to substitute the sand which gives the best results for the sand used by the other Institutes which have taken part in the researches. In the special experiments we shall follow the method of working recommended in your letter as thoroughly suited to the investigation."

Finally Mr. H. Le Chatelier wrote as follows to the President of the Union under date 17th July 1902:

„We have recently remodelled the conditions for limestone and cement contracts for the ministry of public works. As normal sand we have accepted a sand of mixed grain which contains three different sizes of grain obtained by means of sieves of sheet iron perforated with holes of 0·5, 1·0, 1·5 and 2 mm. I believe it would be better to employ merely the two extreme sizes of grain. The sand employed is that on the shore at Leucate in the department of Aube. The preparation of this sand is carried out in the Testing Institution of the „Ponts and Chaussées, Roads and Bridges". I requested Mr. Mesanger to send you a sample.

Two reporters have been selected to discuss this question in connection with the experiments which have been undertaken. Prof. Belelubsky who will specially devote himself to the results to the completion of experiments which have been undertaken in Russia. The undersigned will occupy himself with the other researches which have come to his knowledge. Such researches in-

volve the expenditure of considerable time until a suitable quartz sand is found and prepared as a normal sand. In this report too it is not possible to do more than give a general view of the question such as can serve as a sign-post for later researches.

The Present Position of the Question.

The question of normal sands for strength tests with binding materials has already been made the subject of an important amount of work in different countries. The first country where a satisfactory result has been obtained is France, where the result has been to abandon on the part of the Testing Institutes the artificially triturated sand of Roule, in order to adopt the sand of the seashore of Leucate. As shewn by the study of this sand by the French Committee on methods of testing in vol. IV, sec. B. p. 67, the first session of this Committee contains a notice by Mr. Durand-Claye on the results on comparative experiments with different sorts of sand which has led to the adoption of a natural mixed grained sand as normal sand.

The most complete comparative examination which is at our disposal is that conducted by Mr. Gary of the Testing Institute in Charlottenburg. This refers particularly to the German normal sand of Freienwalde, natural quartz sand. In the year 1896 Mr. Gary published in the „Communications of the Royal Testing Institute", the result of experiments with three Prussian normal sands from Freienwalde, which were composed of rough sand from two different pits. This examination led to the bestowal of the preference on the Hammerthal sand on account of the greater regularity in the size of the sand grains and the colour of the sand and finally because the sand of this pit contains more small grains, such as pass through sieves of 81 and 120 meshes. In 1898 Mr. Gary completed this examination by the publication of an important notice on the normal sands of various countries. He examined

1. The Freienwalde sand passed between sieves of 60 and 120 meshes, the wires being 38 and 32 mm in diameter.

2. Rhine sand similarly sieved.

3. Austrian normal sand from Lemberg sieved between sieves of 64 and 144 meshes, with wires of 4 and 3 mm.

4. The Swiss normal sand of Luterbach near Solothurn sieved similarly to the foregoing.

5. The Russian normal sand between sieves of 64, 144 and 225 meshes with wires 4, 3, 2 mm; the two different sizes of grain thus obtained were mixed in equal proportions.

6. The Norwegian normal sand, a natural river sand, sieved through sieves of 76, 124 and 265 meshes and the grains thus obtained mixed in equal proportion.

7. The French normal sand of Cherbourg through sieves of 64 and 144 meshes; this sand is an artificially triturated quartz sand.

8. The French normal sand of Leucate; the grains being sieved through sieves with holes of 1·5 and 1 mm which were alone used by Mr. Gary. The proper normal sand is composed of 3 equal proportions of grains which would not pass through sieves perforated with holes of 1·5, 1 and 0·5 mm diameter respectively.

9. An English normal sand does not exist and was represented by Leighton Buzzard sand through sieves of 60 and 120 meshes.

10. In these comparative researches sand was employed as American normal sand consisting of crushed quartz sand of the same size of grain as the Rhine sand mentioned above.

In addition to the Swiss normal sand which contains more than 15% of limestone, all these sands contain but a little amount of carbonate of lime, less in fact than 1%. The size and form of the grain produce an effect upon the weight per liter. The crushed or triturated sands or those with sharp corners are lighter, the heavier sands being those of round grains. The difference between the weight where the sand is loosely poured into the measure and where it is tamped in by shaking the mould is fairly constant in the case of the different sands and varies from 280 to 300 grm. per liter.

The comparative strength experiments were made with Portland cement mortar (or concrete) in the proportion of 1 : 3 and 1 : 5 (proportions by weight) the age being 7, 28 and 84 days. The normal test-pieces were stamped or tamped into the moulds

by means of the Böhme apparatus as used in Germany. The amount of blending water varied from 9 to 10% in the case of the concretes of 1 : 5. The strengths obtained with the other samples of sand taken as the means of experiments for 7, 28 and 84 days are contained in the following table in which the samples of sand are grouped according to diminishing strengths.

Sand	Concrete 1 : 3		Concrete 1 : 5	
	Tension	Pressure	Tension	Pressure
The Rhine	104	114	100	116
France	106	111	113	111
Austria	108	104	117	127
England	98	109	94	120
Germany (Freienwalde) .	100	100	100	100
Switzerland	111	81	113	83
France	110	81	114	83
America	115	66	108	65
Norway	90	80	78	81
Russia	72	85	67	78

From the report of Mr. Gary it appears that such numbers being obtained from experiments with a single cement are not definitive; it is to be desired that they should be verified by similar experiments with other binding materials under similar conditions. The numbers which are given for the French sand from Leucate correspond as already explained to a sand with one size of grain, the admixture of three sizes of grain would have a considerable effect on these results.

In general it can be proved that with concrete, in which sand grains with pointed corners or sand that has been obtained by the crushing of quartz, is used, the strength to resist crushing strain is greater, but the tension strength is less. In the case of the Norwegian and Russian sand samples the high proportion of small grains has diminished the strength.

The Testing Institute in Charlottenburg has continued its enquiry on the normal sand question. In the «Communications of the year 1903» there is a very interesting publication by Prof. Gary on German normal sand in which he continues and

summarises the results of the work he has done in conjunction with the Union of cement manufacturers and supported by the Ministry of Public Instruction. This report described the condition and situation of the Freienwalde sand pit, the regulations which are in force at the Testing Institute of Charlottenburg for preparing normal sand and the precautions taken for supplying it to consumers in lead — sealed sacks. These regulations extend to the size of the grains and their composition and also the purity of the sand.

Great attention has been bestowed on getting rid of the difficulties attaching to the use of woven sieves by replacing them by sieves of perforated sheet iron. Sieves with 60 and 120 meshes per cm^2 were compared with the perforated sheet sieves having holes of 1·35 and 0·775 mm in diameter; in the case of the latter the ratio of the fine grains which the latter allowed to pass is distinctly smaller than in the case of the former in which it is not possible to obtain the meshes all exactly of the same size. The experiments showed that the perforated sieves with round holes were superior.

The research was further extended to the determination of the effect of too fine or too coarse grains; the Commission came to the conclusion as the result of very searching experiment that the normal sand may be permitted to contain 10% of grains which pass through the 120 mesh sieve and 2% of the grains which were retained by a 60 mesh sieve.

Finally after the results of the various experiments have been communicated in regard to the effect of impurities, such as pieces of lignite, roots, distillation products of lignite, and including a description of the methods of preparing the new sand at the Hammerthal pit Freienwalde together with the manner in which this is controlled by the Testing Institute at Charlottenburg.

The impression made by the very conscientious work, from which a very short extract has been here given, is that nothing has been omitted as far as Germany is concerned to obtain a normal sand which will satisfy the most exacting requirements as regards equality in the size of grain and purity of composition. In a letter of the 23rd July 1903 Prof. Gary writes as follows:

»We have learnt from the numerous experiments that we had to carry out that even such an extraordinarily equable natural sand as that of Freienwalde does not provide the degree

of equality required by a normal sand. In providing an »international normal sand« the same measures must be adopted as are employed at Freienwalde and it is perhaps worth considering whether Freienwalde might not be advantageously chosen as the source from which an international normal sand should be obtained. At the time experiments are in progress for preparing a mixed grained sand of a higher degree of density. We certainly do not proceed as in other countries to obtain a mixed grained sand by means of three sieves of arbitrarily selected mesh, but we suit the sieves to the qualities of the sand that occurs at Freienwalde, so as to exploit the deposit in the most complete manner. The removal of large quantities of superfluous and rejected constituents is to be avoided and the object aimed at should be to obtain as dense a mixed grained sand as possible which can at any moment be obtained in the required quantity. I think that the experiments can be completed this year so that a report can be furnished to the St. Petersburg Congress.«

The researches for preparing a mixed grained normal sand from the Freienwalde quartz sand have been published by Prof. M. Gary in vol. IV of the »Communications of the Royal Technical Testing Institute in Berlin« for 1903. Though these researches depend on different principles from those recommended by the Comittee of the International Union, they are nevertheless of great interest and cannot fail to be of assistance in solving the question of procuring a mixed grained normal sand. They may be summed up as follows:

The problem which the Committee for normal sand was asked to solve by the Union of German Portland Cement Manufacturers, involves the search for a mixed-grained sand to be obtained by the mixture of German normal sand with residues or waste from the manufacture of this sand. The grains which refuse to pass through a 60 mesh sieve were thrown away, (the amount being too small to be employed) and only what might go through a 120 mesh sieve was used. The waste in Freienwalde contains 24·2% of grains which refuse to pass through the more accurate 120 mesh sieve of the Testing Institute. In addition those 58·7% of grains which refused to pass through the 600 mesh sieve and 17·1% of dust which passed through this sieve. The residue between the 600 and 900 mesh sieve only amounted to 1·6%.

Experiments were undertaken for the purpose of determining the amount of fine sand which mixed with the normal sand would give the greatest degree of density for a mixed-grain sand. An addition of 50% of fine sand to the normal sand raises the weight per liter of the latter which is 1·684 to 1·772 (mixture e) and 1·752 (mixture k) according as the sand residue (the fine sand) was freed from the normal sand grains it contained or not. The mixed sand obtained in this way contains 34% of space cavities. Its density can be still further increased by adding sand dust obtained by sieving the waste through a 900 mesh sieve. An addition of 50% of this dust to the mixed sand produced a sand *q* with a liter weight of 1·917 kg in which 27·6% of space cavity still occurs. The mixture of the normal sand with 50% dust passed through the 900 mesh sieve produces a sand *c* with still greater weight per liter, viz., 1·931 kg. A Portland cement was examined after 7 and 28 days which had been mixed with the sands *n*, *q* and *c*. The mean absolute relative strength figures from 10 experiments with tamped in normal concrete mortar 1 : 3 are as follows:

Sand	Tension			Pressure			
7 days	18·3	19·7	21·0	146	172	175	kg/cm²
28 days	23·9	27·8	28·0	217	238	237	„
7 days	100	108	115	100	110	112	„
28 days	100	116	117	100	110	109	„

The differences are too small according to Prof. Gary's view to justify the difficult preparation of the sand that was employed in these experiments. In order to make the difference in the strength corresponding to the use of the sands of different densities more apparent, it would be advantageous to vary the amount of binding material used. Under all circumstances a mixed grain sand which contains fine dust would give rise to inconvenience in use, particularly on account of the difficulty of obtaining a uniform quality of dust and mixing it equably with the normal sand. The employment of such a mixed grained sand presents no advantages as compared with the usual normal sand.

Although similar experiments have been undertaken in other countries the results have not been published or have not come

to our knowledge. It is, however, advisable to refer to the experiments carried out in Italy for the purpose of providing the country with a uniform normal sand to support of which the Government contributed. These experiments were carried out in a number of different Testing Institutes and with sands of various origin, in accordance with the proposals of the Union Committee.

The experiments carried out at the Testing Institute at Zurich with the Benken quartz sand are reported in the sequel.

To sum up the remarks that have been made above, the position of the enquiry for a sand conforming to the prescribed conditions cannot yet be regarded as concluded. Most of the countries are occupied with the first stages of the enquiry, so that an exchange of the different sands among the different Testing Institutes has not yet been carried out. Only France possesses at the present date a mixed grained sand which corresponds to the prescribed condition; as, however, the strength tests in that country were carried out with test-pieces made in the plastic state the numbers obtained cannot be compared with those of other countries where the recommendations for tamping the concrete into the moulds in a dry condition have been carried out. In order to make an adequate comparison it would be necessary to expand the enquiry to samples prepared by both methods (i. e. in the plastic condition and by tamping the concrete into the moulds).

A number of preliminary experiments in this direction have been carried out by the Testing Institute of Charlottenburg and have shewn that in the case of the test-pieces of Portland-cement concrete prepared in the plastic condition, the strength is considerably less than that of the test-pieces which have been tamped into the moulds. The former were mixed with 13·5% of water, the second with 8·5% on an average. In addition the differences between the individual strengths and the mean is greater in the case of the plastic than in the case of the tamped-in concrete test-pieces. The experiments with plastic concrete compared with the others, possessed the one important advantage that the test-pieces were very much easier to prepare.

The question of enquiring for a uniform normal sand meets with a double difficulty in the way of its solution.

1. In most countries new sources of sand with the required qualities must be discovered.

2. The relative strength figures with concrete of mixed-grain sand have yet to be settled; the existing figures refer only to concretes made with the sand of one particular size of grain.

This double difficulty has been the cause of the slow progress of the enquiry.

Experiments of the tests of the Institute for Testing Materials at Zürich.

Owing to its high percentage of lime-stone, the Swiss normal sand does not fulfil the conditions required for an international normal sand. In addition, the supply obtainable from the pit at Luterbach is very small and can scarcely supply the requirements of the country. Various sorts of quartz sand were proposed, but the purest of them from the Jura have an inadequate size of grain.

A single quartz sand has been found which almost corresponds with the requirements of the Committee, this is the sand of Benken (Canton Zurich).

According to the geological examination of Dr. Rollier, this sand forms the lower portion of the hill called «Brodkorp» in the neighbourhood of Benken. It occurs in horrizontal strata containing interbedded Nagelflue gravels formed by the denudation of the Nagelflue. The deposit can be divided into five strata.

The lowest stratum No. 1 is about 1 m thick and contains whitish yellow sand fairly pure and in places yellow brown.

Stratum No. 2, 50 c.m. thick is a banded sandy marl of grey blue colour.

Stratum No. 3, 1 m thick is formed of coarser sand of variable size of grain and yellow colour.

Stratum No. 5, the thickest is 6 m. thick. It consists of a reddish-yellow sand with smaller and larger pebble or gravel interbeeding of the variegated «Nagelflue». This stratum is the most suited for obtaining the sand.

The highest stratum No. 5, consists of a grey marly calcareous, molasse sand with molasse conretions.

The chemical analyses of the sand gave the following results:

Silica	= 95·2 %
Alumina	= 1·0 %
Iron	= 1·4 %
Magnesia	= 0·8 %
Carbonate of lime	= Trace
Water	= 0·3 %
Alkalis and res.	= 1·3 %

The weight per liter of the sand both when shaken into the measures and not shaken after sieving is:

1. After passing through sieves with 64 and 144 meshes per cm^2 as Swiss normal sand.

2. Through sheet iron sieves *a* and *b* with 2·0 and 1·5 mm diameter holes (sand *b*).

3. Through the sheet iron sieves *b* and *c* (1·5 and 1·0 mm sand *c*).

4. Through the sheet iron sieves *c* and *d* (1·0 and 1·5 mm sand *d*).

The liter weight was also determined for mixtures in equal proportions by weight of sands *b* and *d* and of sands *b*, *c d*. The results obtained are as follows:

	Liter weight	
	Loose	Shaken in
Swiss normal sand	1·420	1·650
Benken sand of equal grain	1·437	1·655
« « *b*	1·454	1·695
« « *c*	1·444	1·685
« « *d*	1·427	1·655
« « *b d*	1·502	1·676
« « *b c d*	1·462	1·655

The form of the grains is shown in the photographs fig. 1 to fig. 5, from which it will be seen that the Benken sand has less sharp corners than the sand from Luterbach.

The crude sand contains about 41 % of sand of the same grain as the Swiss normal sand, smaller proportions of coarser sand and a considerable amount of finer sand *d*. If it is necessary to prepare a mixed grain of normal sand from a larger quantity

of Benken sand the result would be to give rise to a large proportion of waste owing to the greater amount of sieving involved, which is disadvantageous.

In the case of the preliminary experiments which are described in the sequel, the crude sand was sieved and mixed in the Testing Institute at Zürich. The sieves employed are those obtainable in commerce. The sieves *a*, *b*, *c* are of sheet iron, the sieve *d* of sheet brass. The diameter of the holes does not exactly correspond to the size prescribed, for *a* and *b* it was somewhat (about 5 %) too large. For the sieve *c* it was about 5 % too small; only the sieve *d* had holes of the prescribed diameter.

The sieving by means of sieves *a*, *b* and *c* takes place easily, the sieving with sieve *d* on the other hand, owing to the small size of the holes, consumes a great deal of time. In spite of great care the residue left on sieve *d* always contains grains which will only pass through this sieve after prolonged sieving. This source of trouble is always present in the case of a crude sand which contains a number of fine grains, like the Benken sand.

The strength tests were carried out by mixing the sand with various cements and hydraulic lime, so that comparative figures could be obtained.

1. Between concretes with Swiss normal sand and with Benken sand of the same grain.

2. Between concretes with Swiss normal sand and mixed grain Benken sand of two sizes of grain b d and of three sizes of grain b, c, d.

These experiments have been carried out by means of tamping or stamping in the test-pieces with the Klebe-Tetmajer ram apparatus, with which water was allowed to percolate sound the test-piece between the ninetieth and hundredth stroke of the ram. The figures obtained for each cement were got from tests with 6 test-pieces, the mean of the four highest figures being taken.

It appeared advisable to carry out a certain number of tests with plastic concrete (grouting). For this purpose prisms of 4 × 16 cm. were employed as test-pieces in accordance with Feret's method. The six prisms of the series were made in a

mould with 6 divisions or parts. The amount of water employed was so selected that only light tamping was needed to ensure each part of the mould being thoroughly well filled. The prisms were tested by a constant load for deflection between points of support 10 cm apart. Half of the test-pieces were subjected to crushing tests between steel plates of 4 cm in breadth. The tractive strength which is given in the table of experiments was obtained from the bending strength by dividing by 2 in accordance with the usual formula.

Table No. 1 contains the results of the comparative experiments with 5 Portland cements, 3 slag cements and 2 hydraulic limes with Swiss normal sand and with Benken sand of the same size of grain from strata No. 1 and 4 of the pit, after 7 and 28 days respectively. From the comparison of the relative strengths by which that of concrete with Swiss normal sand was taken at 100, considerable differences between one binding material and another, were shewn to obtain. If the means are calculated we obtain the following values.

	Tension strength				Crushing strength			
	7 days		28 days		7 days		28 days	
Sand from Benken	1	4	1	4	1	4	1	4
Portland cements	90	90	93	93	90	94	94	95
Slag cements	94	95	94	91	101	101	100	103
Hydraulic limes	96	100	97	94	104	105	104	100

A great quantity of fine grains, i. e. those which are nearer to the 144 mesh sieve than the 64 mesh in the Benken sand, explains the slight backward tendency in the strengths in this table.

In table 2 the results of a second series of experiments are given with 11 Portland cements, 2 slag cements, 1 Roman cement and 10 hydraulic limes; on the one hand with Swiss normal sand and on the other Benken sand of equal grain in two separate groups, so as to enable any errors that occur in dealing with that particular sand to be eliminated. In these experiments all the test-pieces were examined after 28 days.

The mean value of the relative strengths compared with the Swiss normal sand taken as 100 are as follows.

Sand from Benken	Tension strength			Crushing strength		
	I	II	mean	I	II	mean
Portland cements . . .	90	91	90	95	95	95
Mixed cements . . .	84	87	86	101	103	102
Roman cements . . .	80	76	78	98	94	96
Hydraulic limes . . .	95	93	94	101	101	101

In the tables No. 3 and 4 the comparative results of the tests with 9 Portland cements, and 3 hydraulic limes are juxtaposed. These were made on one hand with Swiss normal sand on the other with mixed grain sand b d and b c d. The strengths were determined after 7 and 28 days and 1 year.

As before the mean relative strengths were determined, that is the strengths of the concrete 1—3 with Swiss normal sand as unity taken as 100, the following strengths being obtained:

	Tension strength					
	7 days		28 days		1 year	
Sand from Benken . .	b d	b c d	b d	b c d	b d	b c d
Portland cements . .	116	108	112	105	90	90
Hydraulic limes . . .	126	118	116	108	106	100

	Crushing strength					
	7 days		28 days		1 year	
Sand from Benken . .	b d	b c d	b d	b c d	b d	b c d
Portland cements . .	134	127	134	125	129	126
Hydraulic limes . . .	122	120	119	121	126	124

These results prove the essential increase in tractive and crushing strength produced by the employement of mixed grain sand. The various tests with Portland cement also show the advantage which is obtained for the purpose of obtaining higher strength when the sand is composed ot two sizes of grain differing very much from one another.

It is possible to compare the numbers obtained with one another so as to determine the relation for Portland cement between the relative strengths of the concrete and Benken sand of a single size of grain (between sieves of 64 and 114 mesh) and a mixed grain Benken sand b d and b c d.

Sand from Benken	64—144	b d	b c d	64—144	b d	b c d
	7 days			28 days		
Strengths of Swiss normal sand = 100						
Portland cement	Tension strength					
	90	116	108	90	112	105
	Crushing strength					
	92	134	127	95	134	125
Strengths of Benken sand (64—144 mesh) = 100						
Portland cement	Tension strength					
	100	129	120	100	124	117
	Crushing strength					
	100	146	138	100	141	131

The increase in strength is markedly greater in the case of the crushing tests than the tractive tests.

The results of the tests with plastic concretes by means of prisms are juxtaposed in tables No. 5 and 6. The prisms were made both with Swiss normal sand and with the mixed grain Benken sand with two and three sizes of grain b d and b c d. The cements and limes employed were the same as in table 3.

The mean relative strengths are as follows, that with Swiss normal sand being taken at 100:

	Tension strength					
	7 days		28 days		1 year	
Sand from Benken .	b d	b c d	b d	b c d	b d	b c d
Portland cements . .	120	122	114	116	95	95
Hydraulic limes . . .	144	126	122	110	108	104

	Crushing strength					
	7 days		28 days		1 year	
Sand from Benken .	b d	b c d	b d	b c d	b d	b c d
Portland cements . .	143	143	140	136	126	127
Hydraulic limes . . .	144	114	125	122	127	125

The results of the tests with hydraulic lime after 7 days are abnormal.

It is of interest to compare the strengths with the tamped-in and plastic concretes with one another; as the test-pieces were not of the same shape this comparison can only be approximate. This was only done in the case of Portland cement between the figures which had been obtained with Swiss normal sand. The results obtained are as follows, the strength of the tamped-in concrete being taken at 100

	Tension strength kg/cm²	
	7 days	28 days
Normal concrete tamped-in	22·7 (100%)	28·9 (100%)
Plastic concrete, prisms . .	14·9 (66%)	20·3 (70%)

	Crushing strength kg/cm²	
	7 days	28 days
Normal concrete tamped-in	243 (100%)	318 (100%)
Plastic concrete, prisms .	134 (55%)	204 (64%)

The ratios of the strengths after 7 and 28 days are:

Tension strength		Crushing strength
Normal concrete tamped-in	1·27	1·30 normal shape
Plastic normal concrete	1·36	1·52 prisms

The results of the density of the test-pieces previously summarised vary little with binding material of the same category. It will therefore be sufficient to give the figures for the density and the corresponding amount of water.

Table No. 1 mortar 1—3 tamped-in; the amount of water for the Swiss normal sand and for the Benken sand of the same size of grain was $9^1/_2$ for the Portland cements, 11% for the slag cements and $11^3/_4$ for the hydraulic lime.

Density of the test-pieces. Tension strength

	7 days		28 days	
Sand:	normal	Benken	normal	Benken
Portland cement . . .	2·37	2·34	2·38	2·35
Slag cement	2·33	2·30	2·34	2·31
Hydr. lime	2·31	2·29	2·32	2·30

Density of the test-pieces. Tension strength.

	7 days		28 days	
Sand:	normal	Benken	normal	Benken
Portland cement . . .	2·39	2·37	2·40	2·37
Slag cement	2·35	2·35	2·35	2·35
Hydr. lime	2·32	2·32	2·33	2·33

Table Nr. 2. concrete 1 : 3 tamped-in, amount of water for the Swiss normal sand with cement $9^1/_2$ with lime 12% for the Benken sand of the same grain $8^3/_4$ for cement and $11^1/_4$% for lime.

Density of the test-pieces

	Tension strength		Crushing strength	
Sand:	normal	Benken	normal	Benken
Portland cements . . .	2·40	2·35	2·43	2·40
Hydr. limes	2·30	2·27	2·32	2·30

Table No. 3 and 4, concrete 1—3 tamped-in; amount of water average.

	Portland cements	Hydr. limes
Swiss normal sand (N)	9·6%	9·7%
Benken sand bd and bcd . .	8·0%	10·1%

Density of the test-pieces. Tension strength

	7 days			28 days		
Sand:	N	bd	bcd	N	bd	bcd
Portland cements	2·37	2·43	2·43	2·39	2·44	2·43
Hydr. limes	2·31	2·34	2·33	2·32	2·35	2·36

Density of the test-pieces. Crushing strength

	7 days			28 days		
Sand:	N	bd	bcd	N	bd	bcd
Portland cement	2·39	2·46	2·45	2·40	2·47	2·46
Hydr. limes	2·33	2·36	2·36	2·34	2·37	2·37

Tables Nos. 5 and 6 concrete 1—3 plastic; amount of water

	Portland cements	Hydr. limes
Swiss normal sand (N)	11·8%	14·5%
Benken sand bd and bcd . . .	9·6%	12·8%

Density of the test-pieces (Prisms)

	7 days			28 days		
Sand:	N	bd	bcd	N	bd	bcd
Portland cements	2·22	2·32	2·31	2·24	2·34	2·33
Hydr. limes	2·14	2·23	2·23	2·16	2·24	2·24

The results which are here communicated although they are not numerous, will suffice to bring into prominence the essential differences between the relative strengths which various kinds of standard sand give with the different binding materials which have been subjected to examination. One can in fact ask the question, whether these differences do not make the search for a uniform normal sand illusory, that is to say a normal sand possessing the quality of giving similar relative strengths with any binding material, if the strength of mortar with the standard sand used is considered as unity. It must be a matter for these who have to carry out the experiments suggested by the Council of the International Association, to perform a sufficient number of experiments so as to determine the limits within which the relative strengths of one binding material compared with another can be established with sufficient accuracy to solve the problem proposed.

The researches which have been communicated show that the exchange of proposed normal sand between the Testing Institutes would not lead to unexceptionable results. It is of greatest importance that these sorts of sands should be tested with the same binding materials, which ought also to be exchanged between the Testing Institutes.

It is clear that only by similar and sufficiently extensive experiments is it possible to determine the characteristics of the sands in different countries which can be recommended as normal sand. It appears under the circumstances, to be advisable that the number of sands proposed for the manufacture of normal sands should not be increased and that the greater number of experiments and researches should be confined to well known types. The amount of normal sand which is required in a country is not very considerable; in most countries the preparation of the sand would require organisation and plant which would come too expensive for the amount to be produced. It would be advantageous for such countries to procure the normal sand from those which already possess the necessary organisation and machinery for preparing it.

I take the liberty at the conclusion of the preliminary report to express the wish that on the occasion of the meeting of the Congress at St. Petersburg a definite list of the proposed varieties of sand should be drawn up, and that future researches should be concentrated on those kinds of sand which most correspond in their qualities, size of grain, purity and the organisation which is employed for preparing them, to the requirements set forth in the circular letter of the Committee.

Enlargement 10 : 1.

Figure 1. **Swiss normal sand of Luterbach**
between 2 sieves 64 and 144 meshes/cm².

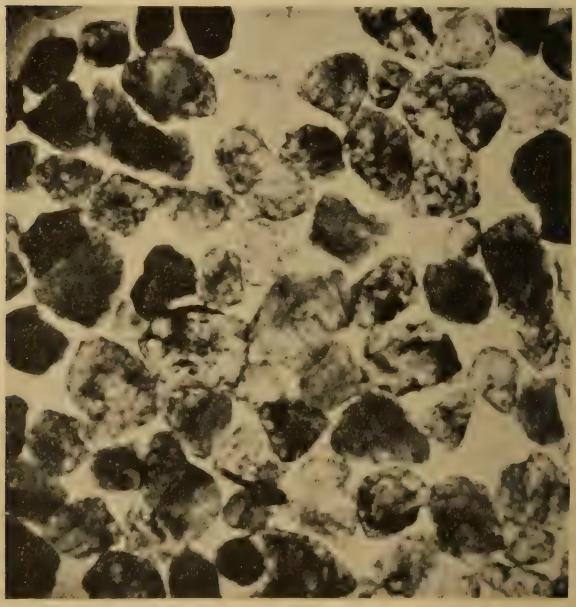

Figure 2. **Benken quartz sand**
between 2 sieves 64 and 144 meshes/cm².

Enlargement 10 : 1.

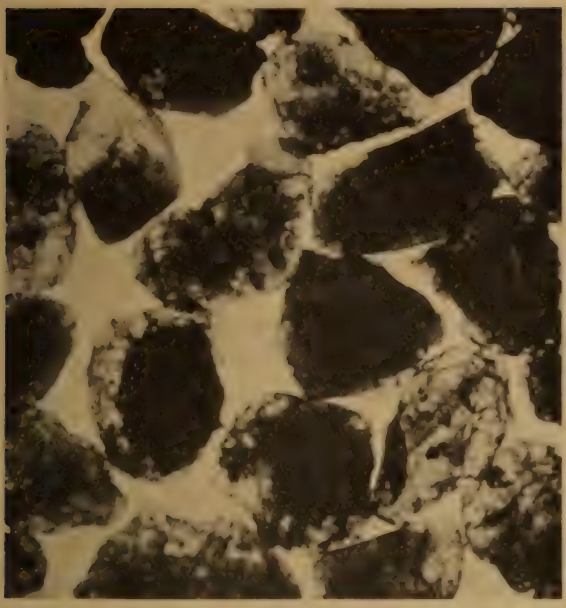

Figure 3. **Benken quartz sand b**
between the 2 sieves perforated a and b.

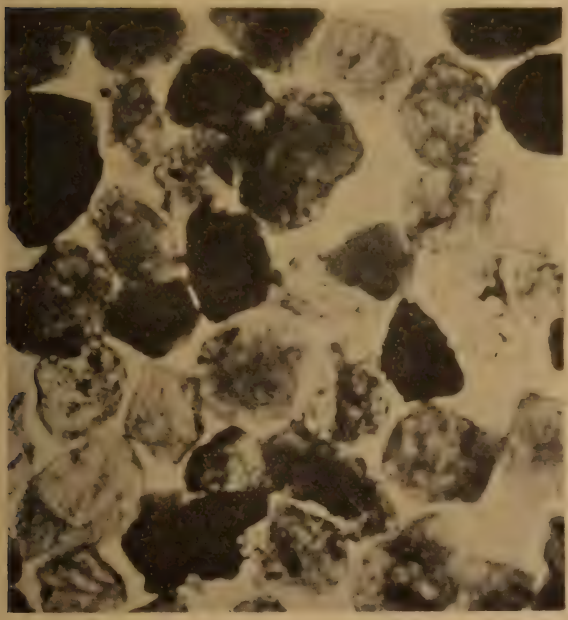

Figure 4. **Benken quartz sand c**
between the 2 sieves perforated b and c.

Enlargement 10 : 1.

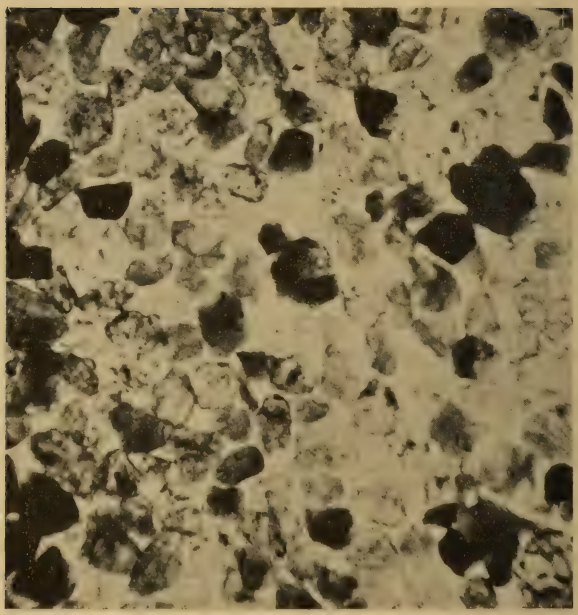

Figure 5. **Benken quartz sand d**
between the two sieves perforated c and d.

Table No. 1.

Results of the strength tests of concretes 1:3 and various binding materials with Swiss normal sand and Benken quartz sand of equal grain.

1st series.

No. of the binding material	Tension strength after 7 days			Tension strength after 28 days			Crushing strength after 7 days			Crushing strength after 28 days		
	sand			sand			sand			sand		
	normal	Benken No. 1	Benken No. 4	normal	Benken No. 1	Benken No. 4	normal	Benken No. 1	Benken No. 4	normal	Benken No. 1	Benken No. 4
1. Portland cements.												
50	20·2	16·2	—	23·5	23·0	—	197·0	160·8	—	249·8	220·0	—
53	20·4	16·9	16·0	27·8	22·6	25·0	198·5	174·8	180·5	269·5	242·3	252·8
54	21·1	20·2	19·4	26·1	24·9	26·4	238·8	227·4	209·9	320·0	322·8	309·8
59	19·0	18·5	—	28·5	27·5	—	215·5	193·8	—	300·5	282·5	—
83	20·3	17·4	20·3	27·7	26·1	24·3	238·8	229·5	249·5	352·8	345·5	340·0
2. Slag cements.												
52	19·9	19·1	19·1	26·3	25·8	24·7	148·3	146·3	145·9	216·5	215·5	221·3
57	11·8	10·6	—	16·3	14·0	—	88·3	90·5	—	111·8	112·5	—
65	16·0	15·6	15·1	23·0	22·2	21·5	122·8	126·5	126·0	169·5	171·3	178·3
3. Hydraulic limes.												
34	3·2	3·2	3·6	9·5	8·6	8·3	24·8	25·6	25·3	57·5	58·8	52·5
71	12·3	12·1	13·2	17·5	18·2	17·4	102·5	106·0	112·0	153·5	162·3	167·3
Relative strengths.												
1. Portland cements.												
50	100	80	—	100	98	—	100	82	—	100	88	—
53	100	83	78	100	81	90	100	88	91	100	90	94
54	100	96	92	100	95	101	100	95	88	100	101	96
59	100	98	—	100	96	—	100	90	—	100	94	—
83	100	85	100	100	95	88	100	96	104	100	98	96
2. Slag cements.												
52	100	96	96	100	98	88	100	99	99	100	99	102
57	100	90	—	100	86	—	100	101	—	100	101	—
65	100	97	95	100	97	94	100	103	102	100	101	105
3. Hydraulic limes.												
34	100	94	94	100	91	87	100	103	102	100	102	91
71	100	98	107	100	104	100	100	104	109	100	106	109

Table No. 2.

Results of the strength tests of concretes 1:3 and various binding materials with Swiss normal sand and Benken quartz sand of size of grain.

2nd series.

No. of the binding material	Tension strength after 28 days			Tension strength after 28 days			Crushing strength after 28 days			Crushing strength after 28 days		
	sand			sand			sand			sand		
	normal	Benken I	Benken II	normal	Benken I	Benken II	normal	Benken I	Benken II	normal	Benken I	Benken II
1. Portland cements.												
212	26·2	25·8	25·2	100	99	96	349·0	305·5	322·3	100	87	92
213	25·0	26·0	27·3	100	104	109	330·5	317·8	321·5	100	96	97
216	33 0	27·7	28·4	100	84	86	400·8	390·3	390·5	100	95	95
221	21·1	18·2	20·9	100	87	99	229·8	197·5	194·8	100	86	85
225	31·0	27·5	27·6	100	89	89	295·5	274·8	279·8	100	93	95
226	27·5	22·8	24·5	100	83	89	311·3	296·0	297·0	100	95	95
232	30·5	25·2	26·5	100	83	87	371·5	338·8	322·8	100	91	87
249	29·3	26·8	26·7	100	92	91	396·5	390·0	386·3	100	98	98
250	24·8	21·5	21·2	100	87	85	334·3	325·0	321·5	100	98	97
254	31·0	23·7	24·0	100	76	78	322·8	321·8	318·8	100	100	99
258	18·2	18·7	18·0	100	103	99	202·0	208·8	211·0	100	103	104
2. Mixed cements.												
245	23·2	17·2	18·3	100	74	79	214·5	217·3	222·0	100	101	104
230	13·7	12·9	13·0	100	94	95	146·3	147·8	148·8	100	101	102
3. Roman cements.												
248	18·0	14·4	13·7	100	80	76	244·8	230·8	228·0	100	98	94
4. Hydraulic limes.												
211	12·7	13·6	11·9	100	107	94	77·8	81·0	82·3	100	104	106
220	9 2	7·2	7·7	100	78	84	57·9	55·3	55·0	100	96	95
222	7·7	9·5	9·5	100	124	124	54·0	69·0	70·0	100	128	130
227	9·5	8 2	7·5	100	86	79	51·9	56·3	57·5	100	108	110
233	10·8	9·4	9·8	100	87	91	57·5	54·3	53·5	100	94	93
234	3·4	3·8	3·4	100	112	100	28·5	29·5	30·3	100	104	106
235	1·3	1·1	1·1	100	85	85	14·8	11·5	11·5	100	78	78
237	11·2	9·5	9·5	100	85	85	55·8	50·0	51·4	100	90	92
238	9·1	8·3	8·5	100	91	93	60·0	59·8	58·5	100	100	98
259	10·7	10·5	10·5	100	98	98	72·0	74·5	74·0	100	104	103

Table No. 3.

Results of the tractive strength tests of concretes 1:3 of various binding materials with Swiss normal sand and Benken quartz sand of two sizes of grain (b d) and of three sizes of grain (b c d).

No. of the binding material	After 7 days			After 28 days			After 1 year		
	sand			sand			sand		
	normal	b d	b c d	normal	b d	b c d	normal	b d	b c d
	1. Portland cements.								
68	29·9	29·9	29·9	33·4	35·2	30·9	38·4	40·4	42·9
73	18·7	22·0	21·8	27·5	32·7	29·3	45·0	41·6	47·7
74	20·4	25·2	23·5	28 3	29·9	30·3	40·1	40·5	41·6
87	21·2	26·2	24·0	29·6	32·3	32·1	38·1	34·4	37·0
98	25·1	26·1	24·9	28·6	32·2	30·0	37·1	42·6	39·5
101	18·2	22·0	18·3	25·4	25·2	23·3	39·2	35·8	31·5
107	20·9	25·8	22·5	25·8	32·2	29·9	41·4	40·5	39·2
108	27·7	29·0	28·6	32 9	34·8	33·4	43·5	45·4	44·3
111	22·2	27·8	26·1	28·4	35·2	32·3	38·5	40·5	37·6
	2. Hydraulic limes.								
64	6·4	6·6	6·3	10·7	11·9	11·6	24 3	26·8	26·2
69	1·9	2·6	2·4	3·5	4·1	3·7	18·9	19·7	18·5
86	8·6	11·8	11·2	17·2	20·8	18·8	33·1	34·1	31·9
	Relative strengths.								
	1. Portland cements.								
68	100	100	100	100	105	92	100	105	109
73	100	118	117	100	119	106	100	92	106
74	100	123	115	100	105	107	100	101	104
87	100	124	114	100	109	108	100	88	97
98	100	104	99	100	112	105	100	115	106
101	100	121	100	100	99	92	100	91	80
107	100	124	108	100	125	116	100	98	94
108	100	105	104	100	106	101	100	104	102
111	100	126	118	100	124	114	100	105	98
	2. Hydraulic limes.								
64	100	103	98	100	111	108	100	110	107
69	100	137	126	100	117	106	100	104	98
86	100	137	130	100	121	109	100	103	97

Table No. 4.

Results of the crushing strength tests of concretes 1 : 3 of various binding materials with Swiss normal sand and with Benken quartz sand in two sizes of grain (b d) and in three sizes of grain (b c d).

No. of the binding material	After 7 days			After 28 days			After 1 year		
	sand			sand			sand		
	normal	b d	b c d	normal	b d	b c d	normal	b d	b c d
				1. Portland cements.					
68	365	454	418	428	540	428	584	780	763
73	176	235	240	253	347	332	596	762	782
74	245	340	302	344	481	446	611	755	819
87	216	318	313	309	434	433	461	623	611
98	235	322	302	292	404	386	475	671	642
101	204	250	228	274	346	305	458	594	515
107	223	314	313	291	410	399	486	714	687
108	272	369	353	346	445	427	525	660	667
111	254	320	292	333	437	397	568	709	655
				2. Hydraulic limes.					
64	45	50	50	83	91	102	275	356	352
69	11	13	12	23	26	24	79	93	88
86	79	111	111	144	195	194	392	510	511
				Relative strengths.					
				1. Portland cements.					
68	100	124	115	100	126	100	100	134	130
73	100	134	136	100	137	131	100	128	131
74	100	139	123	100	140	130	100	124	134
87	100	147	145	100	140	140	100	135	133
98	100	137	128	100	138	132	100	141	135
101	100	123	112	100	126	111	100	129	112
107	100	141	140	100	141	137	100	147	142
108	100	136	130	100	129	123	100	126	127
111	100	126	115	100	131	119	100	125	115
				2. Hydraulic limes.					
64	100	111	111	100	110	123	100	129	128
69	100	116	109	100	113	104	100	118	113
86	100	140	140	100	135	135	100	130	130

Table No. 5.

Results of the tension strength tests of plastic concretes 1 : 3 with Swiss normal sand and with Benken quartz sand in two sizes of grain (b d) and in three sizes of grain (b c d) by means of prisms 4 × 4 × 16 and by binding test.

No. of the binding material	After 7 days			After 28 days			After 1 year		
	sand			sand			sand		
	normal	b d	b c d	normal	b d	b c d	normal	b d	b c d
				1. Portland cements.					
68	22·0	21·4	21·4	24·6	26·1	24·2	37·4	37·2	36·3
73	10·6	13·9	16·2	15·4	19·5	23·5	38·2	39·6	40·8
74	14·7	18·0	16·8	20·3	23·0	22·1	34·3	36·6	35·3
87	13·8	17·1	18·4	19·2	24·2	23·6	29·1	33·1	33·8
98	15·9	17·5	17·1	21·1	22·3	22·5	34·6	33·9	33·9
101	11·4	14·1	14·1	16·7	19·8	18·6	30·5	33·2	30·5
107	14·2	16·8	16·5	20·8	22·5	23·7	34·2	36·3	39·7
108	16·1	19·0	19·9	23·7	25·6	26·6	35·4	36·8	37·6
111	15·7	21·2	20·1	21·3	25·3	25·8	35·0	37·7	37·9
				2. Hydraulic limes.					
64	3·3	3·7	3·9	5·2	6·8	5·8	22·1	20·9	21·4
69	0·7	1·4	1·1	1·9	2·2	2·0	9·2	12·0	11·3
86	5·0	6·1	5·2	8·9	10·6	10·0	27·8	27·5	25·9
				Relative strengths.					
				1. Portland cements.					
68	100	97	97	100	106	98	100	99	97
73	100	131	153	100	126	153	100	103	107
74	100	122	114	100	113	108	100	106	103
87	100	124	133	100	126	123	100	114	116
98	100	110	107	100	106	107	100	98	98
101	100	124	124	100	118	112	100	109	100
107	100	118	116	100	108	113	100	106	116
108	100	118	123	100	108	112	100	104	106
111	100	135	128	100	119	121	100	107	108
				2. Hydraulic limes.					
64	100	112	118	100	131	112	100	95	97
69	100	200	157	100	116	105	100	130	123
86	100	122	104	100	119	112	100	99	93

Table No. 6.

Results of the crushing strength tests of plastic concretes 1 : 3 with Swiss normal sand and with Benken quartz sand in two sizes of grain (b d) and in three sizes of grain (b c d) by means of prisms crushed after the bending test.

No. of the binding material	After 7 days			After 28 days			After 1 year		
	sand			sand			sand		
	normal	b d	b c d	normal	b d	b c d	normal	b d	b c d
				1. Portland cements.					
68	193	225	209	305	344	304	427	490	462
73	101	154	172	144	242	242	440	592	616
74	123	169	173	202	311	301	386	526	508
87	138	193	211	189	285	280	317	427	457
98	141	192	186	201	253	268	345	442	479
101	105	146	146	167	208	199	343	392	392
107	127	168	172	193	254	256	359	479	483
108	141	222	212	233	319	292	395	491	480
111	141	247	223	209	328	322	371	509	509
				2. Hydraulic limes.					
64	24	29	29	42	52	53	190	214	233
69	3·5	6·1	3·6	9·4	11·6	11·4	37	54	53
86	41	57	48	77	97	91	286	344	316
				Relative strengths.					
				1. Portland cements					
68	100	117	109	100	113	100	100	115	108
73	100	153	170	100	168	168	100	134	140
74	100	137	140	100	154	149	100	136	132
87	100	139	152	100	151	148	100	135	144
98	100	136	132	100	126	133	100	128	139
101	100	139	139	100	125	119	100	114	114
107	100	132	135	100	131	133	100	134	135
108	100	157	150	100	137	125	100	124	122
111	100	175	158	100	157	154	100	137	137
				2. Hydraulic limes.					
64	100	121	121	100	124	126	100	113	123
69	100	174	103	100	124	121	100	146	143
86	100	139	117	100	126	118	100	121	110

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 30.

Group B

DETERMINATION OF A UNIFORM METHOD FOR THE SEPARATION OF THE FINEST PARTICLES IN PORTLAND CEMENT BY LIQUID AND AIR PROCESSES.

REPORT PRESENTED BY PROFESSOR
M. GARY, GR.-LICHTERFELDE-WEST.
(TRANSLATED BY CH. CAPITO, LONDON.)

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Determination of a standard method for the separation of the finest particles in Portland cement by liquid and air processes.

Report presented by Professor **M. Gary**, Gr.-Lichterfelde-West.

(Translated by Ch. Capito, London.)

In consequence of the resolution passed by the Council of the «International Association for Testing Materials» (October 28th, 1902), a number of public laboratories and testing institutions have been requested to take part in solving problem 30, and at the same time a reply to the following questions was requested:

1. Are you acquainted with a method for determining the quantity, grain-size and weight of the flour of Portland cement, i. e. of the dust which can pass through a sieve of 5000 meshes per cm^2 and which cannot reliably be further sifted by means of gauze-sieves?
2. If your reply is in the affirmative: on what principle is the apparatus worked, and what degree of separation of the flour does it permit of?
3. If your answer is in the negative: would you be willing to assist in making trials with the object of finding the simplest method of determining the flour of a Portland cement on the principle of fluid or air separation?
4. Would you be willing to assist in making trials for the purpose of testing how far a process, suggested by somebody else, is suitable?

All the replies to question 1, were in the negative. Nobody appeared to know of a process by which the flour of Portland cement can be determined. At that time the only place where a process was at work, by which the flour of pulverised materials could be separated by the action of a current of air, was at the Royal Testing-Institution at Charlottenburg.

The apparatus which was used for this purpose was designed by the reporter and Dr. Lindner, a former assistant at the Testing-station. A description of the apparatus will be found further on.

The following authorities have declared themselves willing to assist, unconditionally, in ascertaining a process by which the flour in a Portland cement may be separated:

1. The Confederate Swiss Testing-Institution at the Polytechnic at Zürich.

2. Professor A. Mesnager, Ingénieur des Ponts et Chaussées, 6 rue de la Boetie, Périgneux (Dordogne).

3. The Royal Testing-Institution at Charlottenburg (now the Royal Institution for Testing materials at Groß-Lichterfelde).

No report, however, has been delivered by 1 and 2 up to the beginning of 1905.

Professor Nagy, Chief of the Testing-station of the Technical High School of Budapest, was willing to give his assistance provided the Council of the Budapest Institution would authorize the trial of certain processes.

Professor J. Hannover, Director of the Danish Government's Testing-Institution of Copenhagen, applied to the Association of Scandinavian Cement Manufacturers, desiring to know whether the Association would be willing to bear the expenses of the tests which would be carried out at the Institution, as the latter possessed no grant to buy the apparatus, but would be willing to give time and energy towards the solution of the problem. The Association agreed to grant the request and the trials began in March 1903. The results will be dealt with later on.

The laboratory of the Imperial Institution for Road-Construction (Professor Belelubsky) of St. Petersburg, overloaded with other problems, declined to assist in the matter. Messrs. Koning and

L. Bienfait of Amsterdam held the view that the problem was not of much practical importance with regard to testing Portland cement. Mr. Roussel of Melines also did not consider it necessary to make a determination of the grains which will pass through a sieve of 5000 meshes per cm^2. He is of the opinion, however, that it would be more desirable to limit the methods of testing Portland cement, as much as possible, to such processes as allow of a direct proof of the quality of the cement.

No doubt, the view taken by Mr. Roussel has much in it, from a practical point of view. It does not seem desirable to increase the number of test-methods excessively. On the other hand, the competition which exists in the field of hydraulic binding-agents creates continuously new products whose characteristic qualities become more and more difficult to ascertain by the old testing-methods.

The simple setting-test or ultimate strength-test, in which standard sand is used, proves ineffective when a rapid determination has to te made. Our aim must be directed towards determining, as quickly as possible, the characteristic differences of the similar binding-agents which are sent in for tender. For this purpose, the process of separation of the flour, i. e. chemically the most active portion, offers a valuable aid, as it not only admits of determining the quantity of the fine or finest grains of each binding-agent, but it also offers an opportunity of judging the homogeneousness of the binding-agent.

Mr. W. F. Goreham of Belvedere, England, has designed an apparatus for separating the finest portion of a cement by the action of a current of air; or, in other words, the apparatus permits of extracting the flour, which could not be done by means of sieves. The apparatus is not supposed to replace the sieve-test, but to determine the quantity of flour. For this purpose, a material for comparison is required which should have a wide range in order to obtain the proper standand. It appears that a rather coarsely ground English cement was chosen as standard, as Mr. Goreham states that cement only exceptionally contains more than 50 per cent of flour, and that a good cement should contain at least 40 per cent of the finest flour.

Fig. 1 is a diagram of Goreham's flourometer. It consists chiefly of two tubes R and r, the latter inside the former. The outer zinc-tube R has a diameter of 8 cm and is closed at the

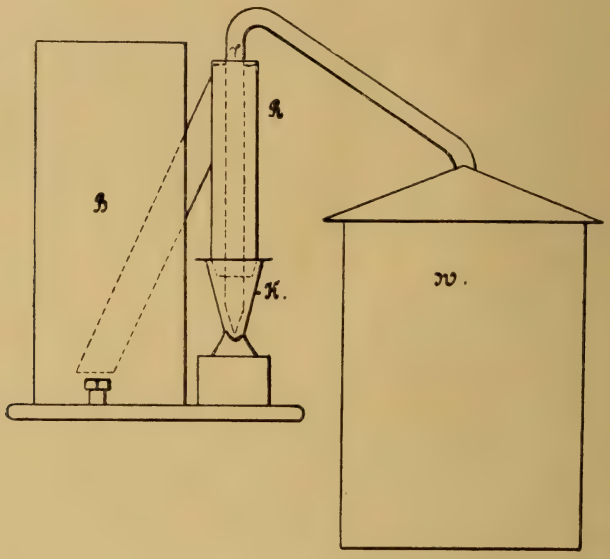

Fig. 1.

top and tapered at the bottom; it fits air-tight into a tapered receptacle in which the cement to be tested is placed. The tube r, placed inside R, is carried air-tight through the cover of tube R and reaches as far as the narrowest part of vessel K. The end-piece is notched so as to prevent the narrow space between the two tubes from being choked with cement. Tube R carries at half its height an uptake-tube which ends inside a large receptacle B. The air is driven through the inner tube from an aërometer W at a fixed pressure and passes through the notches, then it strikes against the tapered sides of the vessel K, and stirs up the cement, of which the receptacle contains about 40 grammes. The flour is carried off by the current of air and the greater portion settles in receptacle B. At the end of the test the cement-residue is weighed.

From a cement which left a residue of 3·2 per cent on a sieve of 5000 meshes per cm^2, and 6·5 per cent on a sieve of 900 meshes per cm^2, there remained 5·1 per cent in the vessel.

From a finer ground cement (leaving residues on the sieves of 2, 4, 5 and 6 per cent respectively) the remainder was only 4·5 per cent. Consistent results are obtained with the same cement.

The flourometer is supposed to be used by the London County Council, the Mersey Docks Board, and by other Associations in Manchester, Liverpool, Birmingham, Nottingham, Plymouth, etc.

Another apparatus, intended for similar purposes, but worked with a liquid-current, has been designed by M. R. Feret of Boulogne-sur-Mer, when investigating the action of grinding and crushing machinery[1]). He points out the difficulty of explaining the figures obtained and their comparison, if the construction and dimensions of the apparatus are not exacty alike.

1. The velocity of a liquid-current is not the same at all points of its cross-section (friction at the surface of the vessel). The size of the grains carried off by the current depends on the maximum velocity, whereas the average velocity only is considered; the relation between the two depends on many circumstances.

2. If the material to be tested contains particles of different specific gravity, then the smallest grain corresponding to a given velocity of current will not be the same for all the particles.

3. On account of the difference in shape and surface-condition, the coarsest grains corresponding to a given velocity of current have neither the same volume, nor the same surface, nor the same linear dimensions; it can only be said that the grains are alike with regard to their floating capacity in the current, and that there exists an altogether unknown relation between their weights and their linear dimensions.

Whatever apparatus we may choose to use, there appears to be no exact relation between the average velocity of the fluid and the volume of the coarsest grains which correspond to this velocity.

For the purpose of turning the results of the trials to useful account, and of comparing them with each other, M. Feret proposes to plot curves with abscissae representing either the

[1]) Baumaterialienkunde 15th December 1903, page 352 and 15th June 1904, page 164.

diameter of the grains (measured under the microscope), or the volume of the grains, or the velocity of the current, and with ordinates representing the weight of the grains.

This curve is useful for the information it may give about the quantity (contained in the tested powder) of grains whose diameter d (or volume v) is larger than any given standard. Starting with ordinate 1 for d (or v) $= o$ the curve approaches the axis of abscissae progressively, and stops at the point which corresponds to the average diameter of the coarsest grains contained in the powder.

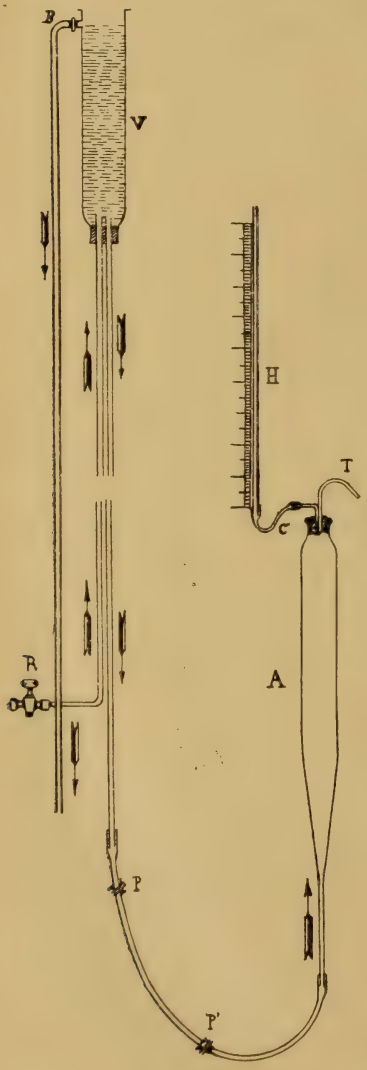

Fig. 2.

M. Feret calls these curves «Integral-Curves». A second curve determined by the angle-coefficients of the temperature of each point in the first curve, is called the «derived curve». The area between the latter curve and the axis of abscissae represents the so-called granulometric structure of the powder.

The apparatus used by M. Feret for making determinations of powders by the above mentioned method (fig. 2.) is similar to Schoene's well-known fluid-separator.

Water flows upwards through a cock, R, into a receptacle, V, in which the water-level is kept constant by an overflow, B. The water flows again upwards from V through a side-tube, A. The cylindrical part of this tube must be long enough to cause the water-particles to flow in perfectly parallel lines in the upper half of

the tube. The side-tube terminates in a neck which is closed by a cork perforated with two holes. The water is led off through wastepipe, T. A graduated tube, which serves as a water-pressure-gauge, is connected to the apparatus by a rubber-tube, C. The increased velocity due to the suddenly reduced cross-section of the current, causes the grains, which have reached the upper part of A, to flow off through T. The material be to tested is placed in A.

P and P_1 are two pinchcocks, the first being used for regulating the water-supply and the second for interrupting the current when testing with uniform velocity. The material which has been separated is freed from the water by draining, and then by drying at 110°; it is afterwards weighed.

M. Feret used the apparatus to make determinations of the coarse grains in raw cement; 25 grammes of cement were used, and the tests were carried out with a velocity of 30 cm per minute. The process has however also been applied for testing finished cements. For further details see M. Feret's communications in the «Baumaterialienkunde».

The question of determining the flour in Portland cement has also engaged the attention of American Engineers. Following up an enquiry from the «Baltimore and Ohio Railroad Co.» of Wheeling, a request was circulated by the periodical «The Engineering Record» with the following results.[1])

Mr. Clifford Richardson separated the finest particles by a treatment with distilled water at 20° C., using a current of air for mixing the powder and the water. (See Hazen, 24th Annual Report, Mass. State Board of Health.)

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[1]) The Engineering Record 1904, Volume 50, No. 8, page 234.

Messrs. Booth, Garrett & Blair used distilled water and a current of air, but without arriving at any useful results.

Mr. Daniel D. Jackson sifted 50 grammes of dry cement through 3 different sieves and treated the finest portion with kerosene oil in an Erlen-Meyer-bulb. The remaining cement was recovered by filtering, and the oil was removed by burning.

The Government Testing-station in Copenhagen applied a current of air in their trials. It was considered inexpedient to use a liquid, as water is hardly suited for the purpose, and other liquids, which do not attack the cement, would be somewhat expensive, as a considerable amount of liquid is required. The Institution, therefore, preferred to use air, and like M. Feret, adopted an apparatus similar to Schoene's funnel. To prevent any jerky changes in the current due to the great mobility of air, it was decided to make the cylindrical part of the funnel considerably longer than in Schoene's apparatus, and thus prevent the cement from being thrown out of the funnel.

Fig. 3.

In the apparatus which was first designed, the cylindrical part was 30 cm long, with a diameter of about 10 cms.; in any other respect, the shape of the funnel was similar to that used by Schoene. The diameter of the lower opening of the funnel was $2^1/_2$ mm.

The funnel was connected by a rubber-tube to the pipe from a centrifugal blower. A pressure-gauge was connected to the blast-pipe through a small side-tube, so that a uniform velocity could be kept up during the different tests.

A number of preliminary trials were carried out with this funnel in the following manner: The funnel was connected to the blower, the valve was opened and 5 grammes of cement were then put in the glass-funnel through a small copper-funnel. A greater quantity of cement cannot be used, as

the lower opening of the glass-funnel would be stopped up. At the end of the trial, the current of air was cut off and the residue taken out of the funnel and weighed; the residue varied between 68 and 64% and the trial lasted for ½ hour to 3 hours. The same cement was used throughout all the trials.

The residue in the funnel and the powder carried off by the current of air were examined under the microscope, and the size of the grains of both powders was measured. It was found that the limit as to diameter was about 0·02 mm.

As the shape of the grains was very irregular, it was difficult to determine the diameter exactly. The above stated diameter of 0·02 mm is the smallest hitherto found.

Three further trials with the same funnel and the same cement were carried out at a later date. After a careful weighing, the residue in the funnel was sifted through a sieve of 5000 meshes on the cm².

The following is the result.

Weight of cement used in separation-test	Duration of separation-test	Remainder in separator-funnel		Residue on a sieve of 5000 meshes per cm²	
grammes	minutes	grammes	per cent	grammes	per cent
5·0	60	3·356	67·1	0·759	15·2
5·0	60	3·359	67·2	0·763	15·3
5·0	60	3·432	68·6	0·839	16·8

The shape of this funnel was not all that might be desired: the lower part was not tapered, but was somewhat suddenly reduced in diameter below the cylindrical part. Another funnel of a more suitable shape was therefore constructed; it is shown in fig. 3.

Trials with this funnel were carried out in the same manner as with the first one. Tests were then made to determine how long the process must be continued till no more dust is carried off. It was established that after a lapse of 15 minutes,

the residue in the funnel was not appreciably diminished. All further trials were therefore only continued for 15 minutes. By holding an incandescent lamp behind the funnel, one can make sure whether any more is carried off, as even small quantities of dust can be detected in the current of air.

Results from a trial with the same cement as before:

Weight of cement used in seperation-test	Duration of separation-test	Remainder in separator-funnel		Residue on a sieve of 5000 meshes per cm²	
grammes	minutes	grammes	per cent	grammes	per cent
5·0	15	2·696	53·9	0·854	17·1

The microscopical examination showed the diameter of the transition-grains between the residue in the separator-funnel and the powder carried off to be about 0·03 mm.

Finally 16 different cements have been tested with the same separator-funnel. These cements were samples taken from the usual run of cements sent in to be tested by the Institution. At least two tests were made with each cement; the results are contained in table I.

In the tests from No. 4 onward, the cement was placed in the funnel in a different manner than hitherto. The cement was weighed in the small cylindrical metal-receptacle shown in fig 4,

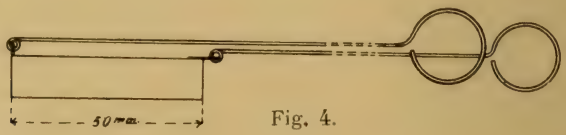

Fig. 4.

which was then placed on the thin wires which may be seen in the figure. After the current of air had been regulated, the receptacle was let down through the upper opening in the funnel, and by the aid of the wires continuously turned till all the cement had fallen out. Having completely emptied the receptacle, it was removed as quickly as possible up through the opening so that no cement dust was lost.

In several of the cases in table I, the remainder in the funnel as well as samples of the dust were examined under the microscope, to make certain that the remainder was freed from all grains of a diameter smaller than 0·03 mm., and also that the dust carried off by the air did not contain grains of a larger diameter than 0·03 mm. It was found that only an insignificant number of grains were not properly separated by this process.

The samples required for the microscopical examinations of the dust had hitherto been extracted by placing a piece of filter-paper across the top-opening of the separator-funnel; a quantity of dust sufficient for the examination would gather on the paper. This method of extracting the material did not however guarantee that particles of larger diameters than 0·03 mm were not carried off simultaneously with the flour.

The following check-tests were therefore made.

The opening at the top of the funnel was ground slightly taper so as to allow a glass-tube to be placed and connected with two bulb-tubes of the shape shown in fig. 5.

In carrying out the tests, the ground tube with the bulb-tubes was placed on the separator-funnel directly after it had been filled with cement. When the test was finished, the powder which had gathered in the different bulbs was extracted, and separately examined under the microscope.

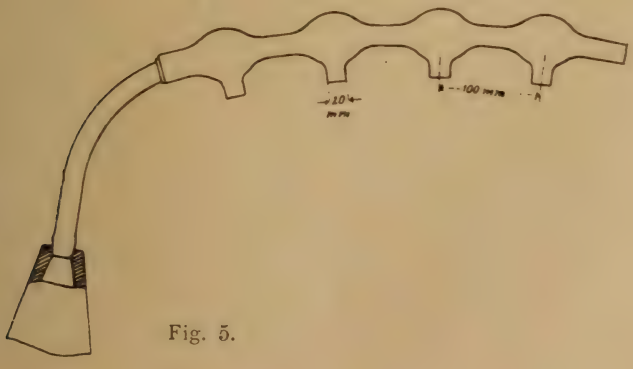

Fig. 5.

No. of test	Weight of cement used in separation-test	Duration of separation-test	Remainder in separator-funnel		Residue on a sieve of 5000 meshes per cm²	
	grammes	minutes	grammes	per cent	grammes	per cent
1 a	5·000	15	2·606	52·1	0·759	15·2
1 b			2·536	50·7	0·733	14·7
2 a	5·000	15	2·070	41·4	0·558	11·2
2 b			2·131	42·6	0·546	10·9
3 a	5·000	15	2·214	44·3	0·543	10·9
3 b			2·282	45·6	0·573	11·5
4 a	5·000	15	2·132	42·6	0·506	10·1
4 b			2·144	42·9	0·513	10·3
4 c			2·108	42·2	0·511	10 2
5 a	5·000	15	2·286	45·7	0·627	12·5
5 b			2·238	44·8	0·696	13·9
6 a	5·000	15	2·789	55·8	1·200	24·0
6 b			2·756	55·1	1·218	24·4
7 a	5·000	15	2·850	57·0	1·284	25·7
7 b			2·879	57·6	1·264	25·3
8 a	5·000	15	2·554	51·1	0·839	16·8
8 b			2·539	50·8	0·834	16·7
9 a	5·000	15	2·167	43·3	0·472	9·4
9 b			2·176	43·5	0·462	9·2
10 a	5·000	15	2·114	42·3	0·569	11·4
10 b			2·087	41·7	0·518	10·4
11 a	5·000	15	2·229	44·6	0·640	12·8
11 b			2·237	44·7	0·626	12·5
12 a	5·000	15	2·525	50·5	0·990	19·8
12 b			2·520	50·4	0·990	19·8
13 a	5·000	15	2·223	44·5	0·635	12·7
13 b			2·206	44·1	0·610	12·2
14 a	5·000	15	2·250	45·0	0·669	13·4
14 b			2·251	45·0	0·670	13·4
15 a	5·000	15	2·013	40·3	0·398	8·0
15 b			2·012	40·2	0·398	8·0
16 a	5·000	15	2·726	54·5	1·178	23·6
16 b			2·640	52·8	1·178	23·6

The results showed that the powder which was caught in the bulb-tubes, and even that in the bulb next to the funnel, only contained a few single grains of a diameter larger than 0.03 mm.*)

Fig. 6 represents a microphotograph of the powder gathered in the bulb next to the funnel. For the purpose of comparison, two microphotographs (same magnification) of the residue left in

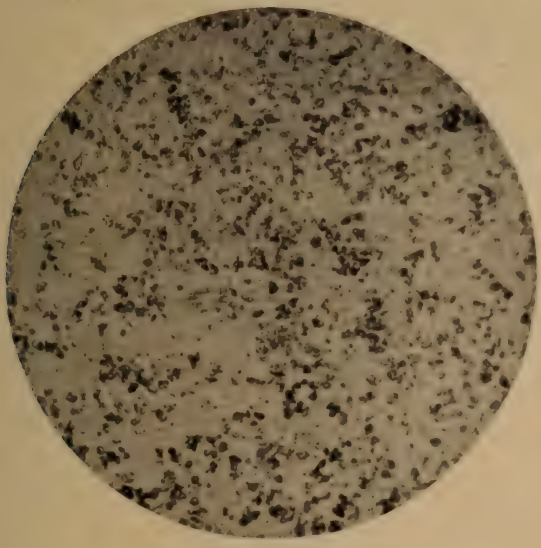

Fig. 6.

the funnel in test No. 1 are subjoined. The first (fig. 7) shows the fine powder which passed through the sieve of 5000 meshes per cm^2, and the second (fig. 8) shows the residue left on the sieve.

The figures given in table I show that various tests of the same cement have given good consistent results.

*) As the bulb-tubes somewhat reduce the velocity of the air, so that fewer larger sized grains will be carried off than in the other case, the residue in the funnel was examined to see whether it was appreciably greater; this was however not the case, but on the contrary the remainder was less. The reason for this must probably be found in the fact that the trial with the bulb-tubes was carried on about 9 months after the first trials, so that the cement, although preserved in a glass-bottle with a glass-stopper, had become finer; it may also partly be due to the difference in degree of fineness, which is observed with different samples from the same cement.

Although some of the tests, viz: Nos. 1, 2, 3 and 16, show a greater discrepancy, the cause is probably not due to any irregularity in the process, but rather to a real difference between the two samples. As a matter of fact, it was amply proved by

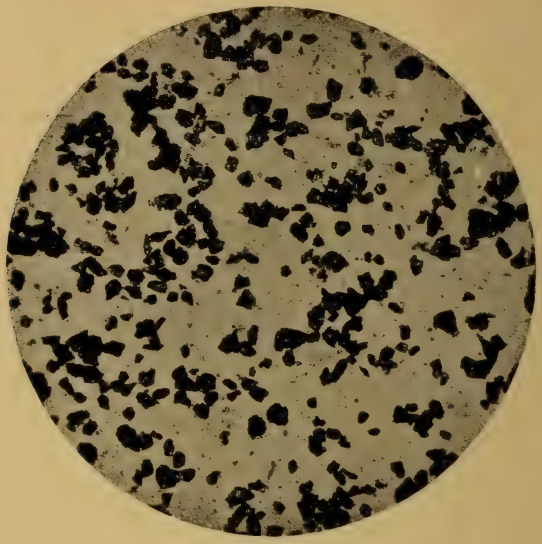

Fig. 7.

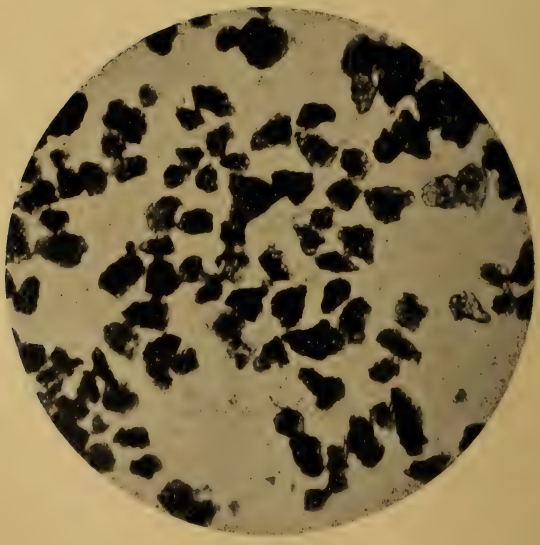

Fig. 8.

two different tests with the same cement, that if the cement, from which the samples were taken, was carefully mixed before the trials, the results were almost exactly alike.

As the trials are not yet finished, the present communication must only be considered as a preliminary report. The Institution intends to arrange for trials with a number of other funnel-separators, for the purpose of deciding on a standard-form which will enable them to obtain consistent results with the same cement. Trials with a funnel of other dimensions, and giving another size of transition-grains between the remainder in the funnel and the portion carried off, are at present being made, but are not yet finished.

The trials with the Gary-Lindner apparatus, which are at present being carried on in the Royal Testing-station at Gross-Lichterfelde, are also not yet finished.

This apparatus, which also depends on the action of a current of air, but aims at more complete granulation, is shown in fig. 9. It consists of three good-sized glass-tubes *a* which are connected with each other and which end in glass-funnels. Small glass-tubes for conveying the air, and extending almost to the bottom, are fused into the funnel-tubes.

20 grammes of the powder to be tested, which must be thoroughly dried beforehand, are placed in the first funnel, I; an air-blast at a pressure of 100 mm (height of water) is then blown in. The air pressure for each funnel is regulated by glass-cocks and is read off on a U-gauge. The funnels I, II and III come successively into operation. Finally a portion of the powder will remain in each funnel, and the flour which arrives as dust at the end of the third glass-tube, is caught in the receptacle IV.

When the air-blast is produced by a water-blower, the air must be dried before entering the funnel; this may be done by inserting a Wolff's flask (with sulphuric acid) in the blast-pipe.

This process admits of a granulation of four fractions of the cement powder, which, as Mr. Feret has already called attention to, are not separated exclusively with regard to the size of the grains, but nevertheless show relative characteristic differences

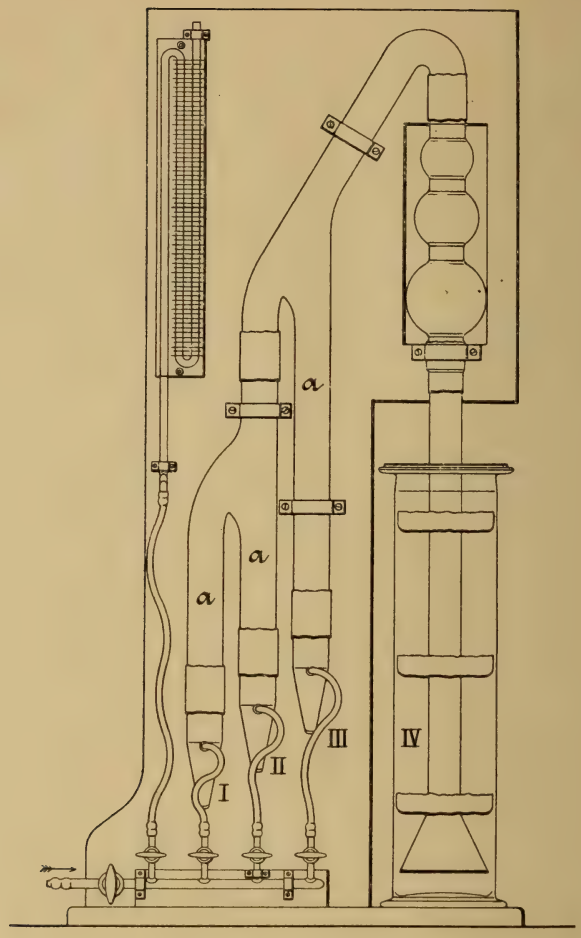

Fig. 9.

and offer new distinctive characters. By this process it will be possible — especially by the aid of the microscope and chemical analysis — to acquire interesting information about the nature of the fine cement flour.

The supposed disadvantage of air-separation which Mr. Feret feared — that it would not be possible to regulate at will, nor measure the velocity of the current of air — is not met with in this apparatus. At all events it is easy to set similar apparatus to the same velocity.

Table II.

Tests of Sidero-Portland-cement with the Gary-Lindner Air-granulator.

Cement direct from the works					Commercial cement				
No. of test	Weight of the residues in grammes				No. of test	Weight of the residues in grammes			
	I	II	III	IV		I	II	III	IV
1	6·77	6·80	2·35	3·25	1	7·55	6·80	2·10	3·06
2	7·52	5·70	2·58	3·50	2	7·58	6·80	2·08	3·05
3	6·90	6·12	2·55	3·25	3	7·22	7·10	2·07	3·05
4	6·33	6·40	2·66	3·50	4	8·05	6·10	2·15	2·95
5	6·85	5·68	2·76	3·95	5	7·66	6·55	2·10	3·05
6	7·21	6·23	2·45	3·20	6	8·00	5·75	2·30	3·25
7	6·80	6·97	2·25	3·05	7	7·63	6·23	2·30	3·07
8	5·95	7·50	2·22	3·16	8	8·15	5·45	2·33	3·41
9	8·17	5·80	2·25	3·25	9	8·23	5·23	2·40	3·36
10	7·73	6·35	2·17	2·80	10	8·63	4·67	2·50	3·40
Sum	70·23	63·55	24·24	32·91	Sum	78·70	60·08	22·33	31·65
Average	7·02	6·36	2·42	3·29	Average	7·87	6·07	2·23	3·17
%	35·10	31·80	12·10	21·00*)	%	39·35	30·35	11·15	19·15*)
Sulphide-sulphur	1·09	1·36	1·18	0·77	Sulphide-sulphur	1·19	1·24	1·22	0·87

*) Included the losses as powder.

Comparison-tests for proving the reliability of the apparatus have shown that, by similar manipulation, consistent results may be obtained.

Table II contains 20 such tests of two cements from the same Sidero-Portland cement works, 10 of which were made with cement direct from the works, and the other 10 with commercial cement. In neither case was the cement carefully mixed before the trial.

The individual results are sufficiently consistent.

The four fractions I—IV (the residues in the funnels) have been tested quantitatively for sulphide-sulphur, and show characteristic differences with regard to this. The separation-process thus proves to be also useful in detecting added blast-furnace-slag.

Table III contains 8 further trials with a Portland cement, a Sidero-Portland cement, a mixture of both and a slag cement.

Table III.

Separation-Tests of two Portland cements, of one mixture of both and of one Slag cement.

Air-pressure = 100 mm height of water. Duration of test = 30 mins. Amount of cement used = 20 grs (dry).

Cement	No. of test	Quantity of grains in per cent by weight in vessel				Loss per cent
		I	II	III	IV	
A Portland cement	1	68·10	11·90	10·35	5·05	4·60
	2	69·40	10·75	10·00	5·35	4·50
B Sidero-Portland cement	1	63·35	1·50	11·65	18·75	4·75
	2	63·00	1·50	13·35	18·00	4·15
Mixture 60 A + 40 B	1	70·05	7·85	6·85	10.60	4·65
	2	72·00	7·05	6·25	10·10	4·60
C Slag cement	1	54·05	11·50	12·75	17·00	4·70
	2	53·85	11·75	12·05	17·60	4·65

The characteristic of the three cements A, B and C determined according to the standard rules, are shown in table IV.

In the sieve-tests, the amount of flour which passed through the finest of the usual sieves was 84, 88 and 92 per cent respectively. The differences between the cements are, according to the sieve-tests, only slight, while the air-separation-tests show distinct characteristic differences.

Table IV.

Characteristics of three kinds of Cements tested with the Gary-Linder Air-granulator.

Mark	Specific density of ignited Cement powders	Loss of Cement powders on ignition	Weight of 1 Litre of Cement		Water in neat Cement		300 grammes of Cement gauged with water to the amount given in last colum		During the determination of the setting time was the average			Test for constancy of Volume according to standards
			poured in	shaken	to the consistency of treacle	for setting test	setting-time a) beginning b) termination of hardening	rise in temperature during setting	Temperature of Air	Temperature of Water	humidity of the atmosphere	
		%	kg	kg	%	%	hrs.	C.°	C.°	C.°	%	
A	3·175 at state of delivery 3·235	1·00	1·279	2·020	30·0	25·0	a) 4 b) 9⅓	0·8	18·5	17·8	60	constant
B	3·046 at state of delivery 2·913	2·90	1·086	1·826	36·0	29·5	a) 3¾ b) 7	5·6	17·0	16·7	70	constant
C	2·858 at state of delivery 3·008		1·145	1·907	30·5	24·7	a) 3 b) 9	2·5	17·5	15·0	64	constant

This is also distinctly noticeable in further trials with other binding-agents and mixtures. (Compare table V.)

Continuation of the table IV.

Mark	Residues from sieve-tests						One part of Cement and 3 parts of Normal Sand by weight						
	meshes on 1 sq. cm.						Water in Mortar-sample	Tensile-test Sectional area at the point of fracture = 5 sq. cms.			Compression-test base-area = 50 sq. cms.		
	5000	900	600	324	240			Tensile-strength in Kgrs. per sq. cm.		Density after ramming	Crushing-strength in Kgrs. per sq. cm.		Density after ramming
	%	%	%	%	%		%	7 days	28 days	$\frac{G}{JM}$	7 days	28 days	$\frac{G}{JM}$
A	16·0	1·6	1·2	0·2	0·0		8·5	24·3	31·4	2·318	243·2	322·8	2·234
B	12·0	0·2	0·0	—	—		8·5	20·4	25·7	2·303	187·8	285·4	2·225
C	8·0	0·2	0·0	—	—		8·5	20·0		2·332	143·2		2·248

Table V.

Air-granulation of four Meals, and Weight per Litre of the Fractions.

Substance employed	Quantity of grains in Vessel in per cent by weight					Density of the individual granulations				
	I	II	III	IV	Loss	Total amount	I	II	III	IV
Portland-cement	62·60	11·25	8·00	12·50	5·65	1·825	1·587	1·548	1·476	1·191
Sidero-Portland-cement	48·00	15·35	11·10	15·85	9·70	1·762	1·659	1·651	1·421	1·206
Portland-cement 50 per cent slag	62·85	9·25	9·50	10·60	7·80	1·683	1·524	1·468	1·437	1·262
Ground granular Blastfurnace slag	57·25	10·20	11·45	13·25	7·85	1·556	1·397	1·429	1·429	1·449

In fig. 10, curves are plotted with ordinates representing the weights per liter of the fractions of the individual meals, and with abscissae representing their proportionate quantities. For each binding-agent we have thus a graphical illustration of the dissimilarity of the particles with regard to weight and degree of fineness.

Fig. 10.

When we succeed — by testing a large number of standard Portland cements — in determining the granulometric curve of an average Portland cement, then such a curve may be used as a standard.

The necessary information must be drawn from further trials, which, for want of time, the Testing-Station has not yet been able to carry out.

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TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 31.

Group B

ON THE BEHAVIOUR OF CEMENTS IN SEA WATER.

A NOTE UPON THE DECOMPOSITION OF CEMENTS IN SEA WATER BY H. LE CHATELIER,

INGÉNIEUR EN CHEF DES MINES, PROFESSEUR AU COLLÈGE DE FRANCE,
PARIS.

TRANSLATED BY F. H. LEEDS, LONDON.

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A Note upon the Decomposition of Cements in Sea Water

by **H. Le Chatelier**, Paris.

(Translated by F. H. Leeds, London.)

The author originally accepted the invitation of The International Association for Testing Materials with the idea of presenting a general review of the investigations that have been carried out upon the decomposition of hydraulic cements in sea water; but on attempting to collate the requisite documents, it was quickly discovered that but little precise information was available upon this important subject, and therefore in the present communication he has decided only to give a sketch of the laboratory experiments he has made himself during the past few years, a full report of which was recently published in the »Annales des Mines«.*)

Mechanical Actions due to Chemical Phenomena.

If chemical reactions are the primary cause of the hardening of cement, it does not necessarily follow that all the reactions which take place amongst the products should have the same effect. On the contrary, there are cases where chemical reactions lead to the disintegration of cement. They may do this in two totally different ways, fraught with very unequal degrees of danger.

*) „Observations préliminaires au Sujet de la Décomposition des Ciments à la Mer" (Preliminary Observations on the Decomposition of Cements in Sea Water). By H. Le Chatelier. Published by Mr. V. Ch. Dunod. 1904.

The reactions may simply bring about a softening of the mass through a formation of compounds having a gelatinous consistency, the cement then losing its hardness and succumbing easily to mechanical agencies which attack it from the exterior—more particularly the action of waves—although its general appearance remains unchanged; or they may be accompanied by swelling and cracking until the material is wholly destroyed. For instance, when lime is slaked (an essentially chemical phenomenon), its hydration causes a swelling and cracking of the entire mass; but the hydration of lime is not the only chemical operation capable of producing similar fissures and swellings, for the action of sulphates upon hydraulic cements gives rise to the same phenomena of disintegration in a much less rapid fashion.

Swelling caused by the Formation of Calcium Sulph-Aluminate.

By introducing important quantities of calcium sulphate into hydraulic cements, it is possible to bring about their disintegration at a more or less rapid speed; and, as a rule, destruction proceeds faster when the material is in the sea than when it lies under fresh water. The proportion of calcium sulphate a cement can bear without failing depends on its nature: cements poor in alumina though containing free lime, such as „ciments de grappier"*), and quick-setting cements containing much alumina but little lime, will tolerate considerable additions of sulphate, up to 10%, without suffering appreciably. Hydraulic limes containing much alumina will not bear more than 1% of calcium sulphate, but those which contain little alumina will bear 5% without falling to pieces. M. Candlot has proved this by preparing mixtures of cements and of hydraulic limes with various proportions of calcium sulphate, making them up into pats, and immersing them, some in fresh water and some in salt.

The author has carried out similar experiments upon mixtures of different hydraulic products with 5 and with 20% of calcium sulphate, the test pieces being kept for a year in a

*) „Ciment de grappier", a term for which there appears to be no English equivalent, is a by-product in the manufacture of hydraulic lime, and is prepared by grinding the residue left unattacked when the bulk of the material is slaked.

saturated solution of calcium sulphate. The following table shows the results obtained. A cross indicates the complete disappearance of the briquette and its conversion into a mud: the figures 0 to 5 represent the relative state of preservation, 0 signifying an entire absence of change, with no cracks and no loss of sharpness at the edges of the test piece.

Nos.	Nature of Cementitious Material	Proportion of Calcium Sulphate	
		5%	20%
	Hydraulic Limes		
29-30	Mussy-sur-Aube	0	0
27-28	Aubigny	✓	✓
13-14	Le Teil: Ordinary lime, not ground	0	0
11-12	„ „ „ ground	0	0
15-16	„ „Ciment de grappier“	0	0
17-18	„ Artificial cement	0	0
	Slag Cement		
37-38	Vitry-le-François	2	5
	Quick-Setting Cement		
25-26	Désiré Michel	×	×
23-24	M. Candlot's Artificial cement	0	0
1-2	Vassy: different beds	4	×
3-4	„	0	×
5-6	„ „ „	2	×
7-8	„ „ „	3	×
9-10	„ mixture of all beds, over-burnt	×	×
	Artificial Portland Cement		
19-20	Boulogne-sur-Mer	2	×
21-22	Mantes	×	×
31-32	Valdonne: well burnt	×	×
33-34	„ brown clinker, under-burnt	×	×
35-36	„ yellow clinker, over-burnt	×	×

The swelling observed in the above described experiments is the result of two opposite actions: 1. the expansion of the calcium sulph-aluminate, 2. the hardening produced by the hydration of the cementitious material: and the phenomena should therefore be rendered much more conspicuous if it were possible to eliminate the action which opposes the swelling. This can be done by making

a mixture of calcium sulphate with the powder of a cement that has previously been completely hydrated, causing the whole to cohere slightly by submitting it to simple pressure. In this way enormous expansions are obtained.

To ensure a complete hydration of the cement, it is first powdered in a mill with porcelain balls until it will entirely pass through a cloth sieve having 4900 meshes per square centimetre, then it is gauged with a large excess of water (at least 50%, by weight), and either kept for several months under water out of contact with atmospheric carbon dioxide, or preferably heated under the same conditions to the temperature of 100° C. for one month. While still damp, the hydrated cement is rubbed down and mixed with half its weight of calcium sulphate, a quantity which is equal to the original weight of the cement taken. The semi-solid paste thus produced is brought into a metal mould, and there exposed to a pressure sufficient to cause it to agglomerate: the necessary pressure varying from 10 to 100 kg per square centimetre. The briquettes obtained by this treatment are strong enough to bear manipulation. They are laid on a strip of filter paper one end of which dips into a vessel of water, the level of the liquid being 10 cm below the briquettes; and the whole apparatus is covered over to prevent evaporation. The dilatation of the test-pieces is measured every day without removing them from their support, for they constantly become more fragile as they swell. The expansion finally becomes very great, sometimes the briquettes attain double their initial size.

At first sight it seems difficult to explain the development of the cracks which grow in cement when calcium sulphate is present. A brittle material like a hard cement should apparently fall to pieces, and should not, on a priori grounds, be able to swell and become distorted without complete rupture. In order to study the mechanism of the production of these cracks, two distinct points have to be examined : 1. the development of the forces involved, 2. the mechanism of the deformation.

The Development of the Disintegrating Forces. The origin of these forces may be traced to the chemical processes involved in the hydration of the lime and in the reaction between the calcium sulphate and the aluminates. The first idea that offers itself for connecting these reactions with the forces set free is to

suppose that the reactions are accompanied by a change in volume. For example: the calcium hydroxide produced by the combination of water with calcium oxide may possess a volume larger than that of its constituents, and the phenomenon of disintegration may resemble that which occurs in rocks under the influence of freezing water. As a matter of fact, the action in question is nothing of the kind, as the author has already demonstrated.

The hydration of lime and of magnesia is accompanied by a considerable amount of contraction, the decrease in volume being approximately one-third of the volume of the water entering into combination. Similarly, a diminution in volume, small but well marked, occurs during the formation of calcium sulph-aluminate. The same thing is to be noticed in all the hydration phenomena of cements. It is easy to recognise this without any measuring instrument. At the moment of setting, the free surface of a briquette becomes suddenly dry, as if the water had evaporated. In reality, the water enters into the pores of the material, being sucked in by the contraction which results from the act of combination

The swelling observed in cements is therefore merely an increase in the apparent volume, but not in the absolute volume of the substance; that is to say, whereas the total volume of the solid matter diminishes, the spaces between the solid particles grow larger more rapidly, so that as a whole the mass exhibits an external increase in volume. The solid crystals of calcium hydroxide or of calcium sulph-aluminate press one against the other, so to speak, and drive each other apart by pushing against the solid bodies with which they come in contact.

Mechanism of the Progressive Deformation.

Having thus traced, as accurately as possible, the derivation of the forces developed by the chemical reactions, it remains to see how, by a sufficiently prolonged action, these forces are able to produce a gradual deformation, a merely partial rupture, in a material which at first sight appears as absolutely brittle and rigid as cement. The explanation of this phenomenon can be given in precise terms, for it depends directly upon the laws of solubility. When a solid body is brought into contact with a liquid in which it is soluble, it dissolves until a certain concentration is attained,

and the concentration is strictly governed by the pressure and the temperature. The solution which remains in equilibrium with the solid is said to be saturated with the body in question, and the limit of solubility, termed the coefficient of solubility of the body, varies with the pressure and the temperature of the system. But when a salt which is in contact with a saturated solution is compressed without the liquid suffering compression, the solubility of the solid increases immediately; it therefore dissolves in the surrounding liquid, and the new solution becomes super-saturated with respect to any neighbouring crystals that are not in a state of compression. The liquid thus begins to deposit crystals, becoming less saturated, and acquiring once again the power of dissolving the compressed crystals. In the case of a porous mass formed of salt crystals and interstitial spaces full of water, the salt dissolves gradually and crystallises in the spaces, so that finally the whole of the latter are filled with salt, the liquid is forced to the exterior of the mass, and the material tends to become absolutely compact. Conversely, it may be imagined that if a porous but coherent mass composed of crystalline grains attached to each other is submitted to a tensile strain, the crystals extended have their solubility increased, dissolving and permitting the mass to be progressively deformed (after the fashion of a malleable substance) until the moment arrives when total rupture takes place.

This is exactly what occurs in cement under the influence of the chemical reactions which give rise to swelling. It is easy to show (especially with plaster of Paris) that porous cements are progressively deformed without rupture under the action of a mechanical load, just as happens with malleable materials. A number of test-bars of plaster of Paris, some dry and some saturated with water, 1 cm^2 in section, were placed on supports 100 mm apart, and loaded until they broke. The results were:

	1.	2.	Mean
Dry bar	7·04 kg	7·125 kg	7.08 kg
Wet bar	3·57 „	3·65 „	3·61 „

A dry bar, and one immersed in a solution of calcium sulphate, were then loaded with half their breaking weights (3·54 and 1·80 kg respectively). The dry bar was still intact after a period of

two months, but the wet bar broke in 24 hours after assuming a permanent deflection of 0·8 mm. Another wet bar was loaded with a quarter of its immediate breaking weight, i. e. 0·90 kg. It broke on the 49th day after taking a deflection of 1 mm. By repeating the experiments upon test-pieces only 5 mm thick and 300 mm long for a period of one month, it was found possible to obtain deflections of 30 mm before rupture. In the latter case the loads employed were, of course, much smaller.

It may appear surprising that similar deformations should be possible in a material like cement whose constituent silicates and aluminates possess such an extremely low degree of solubility. But it must be observed that although the substances are less soluble, their dimensions are smaller, and consequently the area of their surfaces in contact with the liquid is larger; hence, although the speed of dissolution diminishes with the solubility, it increases very rapidly with the superficial area, and therefore by virtue of these two opposing actions the speed of deformation may in all instances be appreciable.

If there be introduced into a cement a substance which tends to develop internal forces by its expansion, a substance like lime or magnesia that suffers hydration, the above described phenomena of progressive deformation may be produced in presence of water. But when once the cement has dried, the free lime or magnesia which is capable of subsequent hydration is generally unable to determine rupture. It could only do so if the particles of quicklime were quite large, such as are never met with in cements, but then the rupture would be complete. Similarly, calcium sulphate, which causes considerable, though very slow, swelling of cement in the damp state, is almost entirely inert if the substances are permitted to dry in air before the reactions have had time to take place. On the other hand, whenever swelling is produced in presence of water through the occurrence of one or other of the above mentioned reactions, its development is progressive as in the experiments where mechanical forces were allowed to act upon the test-bars of plaster of Paris.

This action of expansive forces makes itself manifest by two simultaneous but distinct phenomena: an apparent swelling, i. e. an increase in the total volume, and a formation of cracks. It frequently happens when a set cement is being examined for the

purpose of estimating its deterioration, that only superficial fissures in the briquettes are sought for; yet enormous swelling may sometimes occur without any visible crack. For example, by adding 10% of calcined magnesia in impalpable powder to Portland cement, the author has obtained an increase equal to 30% of the original volume at the end of eight months, although not even the smallest fissure was to be discovered. The development of cracks is the more serious as the swelling is the more rapid, and as the expansive agents are the less uniformly distributed throughout the material. Magnesia in large particles would produce cracking where a fine powder only causes a uniform increase in size.

The Physical Phenomena of the Penetration of Cement by Sea Salts.

The mechanism of the processes whereby the saline matter of sea water penetrates into the interior of cement is of capital importance in studying the spread of decomposition.

Porosity.

The first idea that presents itself is that sea water penetrates into cements by filtration under the influence of differences of pressure brought about by variations in the height of the sea, by the impact of waves, or by the emptying of dry docks. It is perfectly true that a passage of sea water occurs through masonry. This is easy to detect from the streams of water that issue from the walls when there is a difference of level in the liquid on the two sides. But such passage of water only takes place through flaws in the work, through cracks in the cement which joins the stones together. The penetration of water so caused has certainly a great effect upon the permanence of the structure, for it brings the decomposing action of the sea into the heart of the material; but it is quite independent of the nature and of the quality of the hydraulic substance employed. It is obvious that a circulation of liquid through a body of cement itself is almost impossible, for the speed of circulation depends not only upon the total section of the channels open to its passage, but also upon the dimensions

of each of them. The state of subdivision (whether fine or coarse) of the precipitates formed by the action of sea water must cause the size of these channels to vary most irregularly, and so to oppose to the circulation of the liquid a resistance always great but very unequal in amount.

Diffusion.

The diffusion of salts does not meet with the same obstacles as the flow of liquids in porous bodies. Its speed depends solely upon the total free section of the passages, and not upon their individual size. It might be thought, therefore, that diffusion of salts should proceed in a much more regular manner than flow of liquids in bulk; and experiments have shown such to be the case. The method employed by the author to demonstrate this consists in immersing blocks of cement in saline solutions containing a body whose presence in the interior of the mass can be easily detected by appropriate chemical reagents. Among the substances tried, that which has given the best results is potassium ferrocyanide, for the dark blue precipitate produced when it comes into contact with a solution of ferric chloride in hydrochloric acid can be recognised with certainty. A $10^{0}/_{0}$ solution of sodium sulphide may also be used, for it has the property of striking a greenish-black colour wherever it penetrates in the briquette, without the intervention of any other metallic salt.

With the aid of this method it is interesting to study the behaviour of different kinds of hydraulic materials. Briquettes kept for 3 years under lime water were immersed for six months in the $10^{0}/_{0}$ solution of sodium sulphide. In these conditions most hydraulic limes and cements tested were penetrated to their centres, i. e. to a distance of at least 10 mm (the pieces being 20 mm in diameter). A few, however, exhibited only very slight porosity. All the samples, with one or two exceptions shown in the following table, were re-ground before being gauged until they passed a sieve of 4900 meshes. The proportion of water used was always large, generally about $50^{0}/_{0}$, in order to bring the condition of the cement briquettes in the matter of porosity into line with that of the hydraulic limes. In gauging the cements with so large a quantity of water, it was found necessary either to allow them

to stand for a time before the operation was completed, or, more frequently, to start the setting process by heating the paste for a few minutes on the water-bath.

Three years old cements treated with sodium sulphide.

No. of Sample	Nature of hydraulic material	Loss on ignition per 100 parts of residue (Free and combined water)	Depth of penetration of the sulphide into 20 mm cylinders
1	Fat lime, 1 part; burnt clay, 0·5 part	175·7	total
2	English Portland cement	63·5	—
3	Fat lime, 1 part; burnt clay, 1 part .	149·9	—
4	Adulterated Portland cement 1/2 . .	59·9	—
5	» » » 1/1 . .	61·3	—
6	Qick-setting Grenoble cement. . . .	101·2	—
7	Slow-setting » »	83·6	—
8	Boulogne Portland cement	56·75	-
9	Ditto + 1 part of burnt clay . . .	111·3	—
10	Donjeux slag cement	76·1	—
11	Vitry slag cement	82·5	—
12	Fat lime, 1 part; trass, 4 parts . . .	88·4	5 mm
13	Cement of high hydraulic ratio . . .	44·8	0 mm
14	» » » » » . . .	47·0	0 mm
15	Vassy cement, medium grade . . .	91·5	total
16	Teil «Ciment de grappier», not ground	70·0	—
17	Teil hydraulic lime, well burnt . . .	116·8	—
18	Teil «Ciment de grappier»	72·2	—
19	Ditto + 1 part of burnt clay . . .	127·41	—
20	Teil hydraulic lime	73·0	—
21	Ditto + 1 part of calcined silica . .	144·0	—
22	Ditto + 1 part of burnt clay . . .	95·0	1·5 mm

The first column of the table shows the loss in weight on ignition per 100 parts of residue, and represents very nearly the water used in gauging, except in the case of limes containing a noteworthy proportion of combined water. With the exception of No. 18, all the cements were ground before gauging, as mentioned above. Samples 15 and 16 were prepared specially at the Boulogne-factory in order to see whether cements of high hydraulic ratio were better able to resist the decomposing action of sea water.

The slurry had been made with 24·15% of clay. The proportion of water used in gauging was much smaller than in the other samples. These two cements were absolutely impermeable, whereas all the others were porous.

A mixture of Teil hydraulic lime with clay is shown to be almost entirely impermeable, while mixtures of Portland cement or «ciment de grappier» with the same quantity of clay are porous. But in this case the proportion of water was different, viz. 95% in the lime mixtures, of which about 8% already existed in the lime; as against 111 and 127% in the cement mixtures. The experiments quoted in the following table prove that mixtures of cement with clay also become non-porous when they are gauged with less water.

Nature of hydraulic material	Proportion of water	Penetration of sulphide
Lime and grey puzzuolana		8·0 mm
Lime and yellow puzzuolana		3·0
Dèvres cement, well burnt, and grey puzzuolana . .	60 %	total
Ditto yellow „ . .	56	0·5
Dèvres cement, under-burnt, and grey „ . .	54	5·0
Ditto yellow „ . .	60	0·8
Boulogne cement, excess of clay and grey „ . .	51	total
Ditto yellow „ . .	55	1·5
Boulogne cement, excess of lime, and grey „ . .	48	0·5
Ditto yellow „ . .	55	3·0

Finally, a mixture of fat lime with trass was incompletely penetrated.

The above table shows the behaviour of mixtures of lime or cement with Italian puzzuolanas, a yellow and a grey specimen being used for which the author is indebted to M. Rebuffat. Except when mixed with the fat lime, when they were employed in the natural condition, the puzzuolanas were ground like the cements. The cement mixtures were prepared with equal weights of material; the lime mixtures contained 4 parts of puzzuolana to 1 of lime. It will be seen that even when a considerable proportion of water is used, i. e. 50%, hydraulic materials exist which yield absolutely impermeable mixtures.

It would seem that the greater or smaller permeability of hydraulic mortars to the penetration of saline matter by diffusion should be at least as important as differences in the chemical properties of the cements employed when the materials are to be exposed to sea water.

Removal of Lime by Diffusion.

It has been recently pointed out by M. Maynard, Director of the «Laboratoire des Ponts et Chaussées» at La Rochelle, that chemical action is capable of materially altering the porosity of a cement and therefore its permeability to dissolved salts. He has observed that magnesia is never to be found in the interior of hydraulic cement, even when it is in a state of advanced decomposition, provided at least that disintegration is not complete; and yet the lime steadily disappears while the material regularly becomes more and more porous. The lime gradually diffuses from the interior towards the surface, meeting dissolved salts of magnesium which it precipitates.

The author has verified M. Maynard's statement by means of a qualitative process that gives very conspicuous results. Instead of immersing the samples in solutions of magnesium salts, the penetration of which can only be recognised by accurate analysis, he has used coloured salts of silver, mercury, copper, and cobalt. After remaining several months in such solutions, even cements of a relatively high porosity were only coloured to a depth of less than 0·1 mm, i. e., the penetration was practically nil. Here, of course, it is a question of penetration into a compact mass. When the cement cracks and disintegrates, as happens if sulphates of the above mentioned metals (especially the sulphates of copper and cobalt which can be used in a concentrated form) are employed, the liquid enters by all the cracks, and their surfaces become stained; but on breaking the sample, it will be found devoid of colour at all places except the two walls of the fissures.

Impermeable Coatings.

Although the chemical reactions that occur in cement are liable to increase the penetrability of the material, they are also, by a different procedure, capable of reducing its permeability to a considerable extent, and of closing all its pores. Two cases have to be distinguished according as solutions of chlorides or nitrates or solutions of sulphates are employed. In the former case, the superficial layer produced appears to become absolutely impervious; and, having always a high degree of adhesiveness,

causes diffusion from the surface to the saline liquid to proceed very slowly, if indeed it continue at all. When sulphate solutions are used, the phenomena are totally different. The same film is formed; but, after a certain time, it becomes loose, giving rise to blisters, and falling away from the surface of the briquette. The swelling due to the presence of sulphuric acid leads to a complete destruction of the film, and thus allows the material to deteriorate progressively. Hence a fundamental difference must be drawn between solutions containing sulphates and such as do not contain them. In solutions free from sulphates, e. g. that of magnesium chloride, all the experiments that have hitherto been carried out fail to show any decomposition whatever. The protection afforded by a skin of an insoluble metallic oxychloride or oxide appears to be absolute.

Inasmuch as sea water contains chlorides, sulphates, and bicarbonates, the different effects of these salts are produced simultaneously in an unequal fashion, according to the nature of the cements and manner in which they are employed. Very slight modifications in the experimental conditions either cause the film to be permanent and preservative, or prevent its persistence, and greatly accelerate the rapidity of destruction.

It may be interesting to examine the manner in which lime disappears from a hydraulic cement by diffusion towards the exterior. The author has found a reagent, viz. mercuric chloride, that allows the progress of this diffusion to be studied with great accuracy. When a new fracture in a briquette is moistened with a saturated solution (10 %) of mercuric chloride, and, after waiting 10 seconds, the place is washed with plenty of water, all parts of the briquette still containing free lime assume the yellow colour of mercuric oxide, all parts containing only silicates and aluminates of calcium take the reddish-brown colour of mercuric oxychloride, while the parts that have been completely converted into carbonate and decomposed remain colourless. On applying this test to briquettes which had been immersed for a year in solutions of sulphate and chloride of copper or cobalt, the author found the lime not to be completely removed at any point, for their broken surfaces turned reddish-brown except at the spot occupied by the extremely thin superficial layer of basic chlorides and sulphates. The free lime had vanished from a somewhat greater depth but

rarely from more than 1 mm. Thus it is manifest how powerful is the protective agency of the superficial film; for had the same briquettes been soaked even for a much shorter period of time in solutions of soluble salts like ferrocyanide or sodium sulphide which are not precipitated by the cement, they would have been found to be impregnated with the saline matter to a depth of several centimetres.

Mechanism of the Decomposition of Cements. It follows from what has been said that the decomposition of cement is an effect of two successive actions: 1. the removal of lime by diffusion, which destroys the solidity of the material, and increases its porosity, 2. the swelling and disintegration of the weakened mass caused by the attack of calcium sulphate. The first phase of the phenomenon necessarily occurs in two very distinct periods. As long as cement contains free lime, the relatively great solubility of the latter enables it to diffuse rapidly; but when the lime has disappeared, and only silicates and aluminates remain, decomposition is still able to proceed in the same fashion because these bodies dissociate on coming into contact with water. Nevertheless, as the amount of lime liberated by dissociation is extremely minute, its elimination by diffusion is equally slow.

The mechanism of the second phase (decomposition by swelling) is more difficult to understand, for there is no penetration of foreign salts into the cement. It would seem that the cavities and cracks found in an old cement result solely from the stresses set up in the superficial skin by the process of expansion. As long as this skin is not firmly attached to the cement it may be detached without prejudice to the internal resisting power of the material; but when once, through the partial removal of the lime, the film has begun to be precipitated within the pores of the cement, and thus adheres firmly, the strains resulting from the swelling which arises in course of time in the manner already indicated succeed in producing cracks in the cement itself.

If it be admitted — and the author's experiments appear to establish it as a fact — that all the phenomena of decomposition which occur when cements are exposed to sea water (elimination of lime as well as swelling) depend on the action of a little impervious skin, possessing a greater or less power of expansion,

and only a few tenths of a millimetre in thickness at the most, we can at once see why the behaviour of cement on a large scale is apt to appear capricious. Its degree of permanence is governed by the conditions that control the formation and destruction of this skin, which is in a state of perpetual transformation like a living creature, for lime is constantly being brought by diffusion to act upon its inner surface, while the magnesian salts of sea water attack it persistently on the outside. The magnesium compounds destroy the calcium sulph-aluminate which is then formed again either at the same spot or deeper in the body of the cement according to the irregularities in circulation of the lime and of the said magnesium salts.

Conclusion.

The foregoing observations show the following to be the most important conclusions that are to be drawn from a chemical point of view regarding the decomposition of hydraulic cements when exposed to sea water.

1 All the active ingredients in cements: lime, aluminates, and silicates, are decomposed immediately they come into direct contact with the magnesium salts of sea water, yielding soluble chlorides and sulphates of calcium, and so bringing all the lime present into a state of solution.

2. When the calcium sulphate found in natural waters or formed by the interaction of magnesium sulphate and the calcium compounds of cement reacts with calcium aluminate, it produces a calcium sulph-aluminate whose crystallisation gives rise to swelling and cracking in the material. The action resembles that consequent upon the hydration of quicklime, but is much slower.

3. Penetration of marine salts takes place in two different ways: sea water penetrates en masse through all the flaws in the joints of the masonry and through the crevices in the stones and bricks themselves. Most of these flaws in workmanship are unavoidable. From the present aspect, the normal porosity of cement plays only a secondary part in the process.

Afterwards, when the cement is sound, circulation of, and attack by, sea water occurs almost exclusively by a process of diffusion, being the more rapid as the normal porosity of the cement is the greater.

4. All the phenomena of decomposition in sea water are at the mercy of a superficial film of extreme tenuity, whose impermeability tends to prevent, or rather to hinder, diffusive action, but whose expansion, caused by the formation of calcium sulphaluminate, promotes swelling of the material and cracks through which the salt water soon penetrates in quantity.

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REPORT ON THE PROBLEM 32

Group E

ON ACCELERATED TESTS OF THE CONSTANCY OF VOLUME OF CEMENTS

PRESENTED BY THE CHAIRMAN OF
THE COMMITTEE 32

ΩΩ BERTRAM BLOUNT ΩΩ
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The Committee appointed for the solution of Problem 32 consisted of the following members.

G. Greil, Austria,
Th. Pierus, Austria,
E. Hiertz, Belgium,
M. Petersen, Denmark,
Bertram Blount, England,
R. Curling Styles, England,
E. Leduc, France,
F. M. Meyer, Germany,
F. Schott, Germany,
L. Bienfait, Holland
Des. Nagy, Hungary,
J. Zhuk, Hungary,
O. Rebuffat, Italy,
O. Carlsen, Norway,
A. Baykoff, Russia,
O. Blaese, Russia,
E. Schwarz, Russia,
V. Tagneef, Russia,
R. W. Lesley, United States,
S. Newberry, United States.

A meeting of the Committee was held at Brussels on January 26th 1903, which was attended by the members mentioned below:

Bertram Blount (Chairman), England,
E. Hiertz, Belgium,
Mayntz Petersen, Denmark.
R. Curling Styles, England,
E. Leduc, France.
L. Bienfait, Holland.

At this meeting it was decided that some form of hot test should be adopted as a standard accelerated test for the constancy of volume of cement. Experiments were to be made on samples procured from the various countries represented to decide the best temperature for the test and the most suitable means of measuring the expansion of the test pieces. A representative of each country was chosen to carry out the experiments; the following is a list of the members invited to undertake this work.

Alfred Greil, Austria,
Mayntz Petersen, Denmark,
R. Curling Styles, England,
E. Leduc, France,
F. Schott, Germany,
L. Bienfait, Holland,
Des. Nagy, Hungary,
O. Carlsen, Norway,
E. Schwarz, Russia,
R. W. Lesley, United States.

A detailed programme of the methods of carrying out the tests is given in Appendix I.

A report of the resolutions formulated by the meeting at Brussels was communicated to each member ot the Committee and a copy of the programme of work with an invitation to cooperate was sent to the members chosen by the Committee to carry out the experiments.

Requests were sent to members of the Committee and others for samples of cement on which to experiment. The samples were to be (1) sound, (2) doubtful and (3) unsound. It was arranged that portions of each sample should be distributed to those members of the Committee who were willing to undertake the work of experimenting.

A letter was addressed to M. Rebuffat asking him to investigate the nature of the substances which tend to cause the expansion of cement with the view of arriving at a means of identifying and determining them by proximate analysis or microscopically.

Working on the lines laid down above, Mr. Leduc (France)

Mr. Bienfait (Holland), Mr. Schwarz (Russia), Mr. Petersen (Denmark) and Mr. Styles (England) have carried out a series of tests according to the programme and have communicated the results of their experiments. These results are given in Appendix II.

For the convenient discussion of the work done by the Committee a comparative table has been prepared giving the results of tests made on a series of 3 cements by the different observers named above.

These three cements were kindly prepared by a well known firm so as to form a series from the same raw materials and prepared by the same process, differing only in respect of the fact that the first was sound, the second doubtful and the third unsound[1]. They were analysed and tested by Mr. Styles, and were then distributed to all Members of the Committee who had undertaken the investigation of their behaviour when examined according to the programme.

The following are the figures obtained by Mr. Styles:

Chemical Composition.

	1 A, Sound. %	2 A. Doubtful. %	3 A. Unsound. %
Silica	21.76	21.64	21.92
Insoluble residue	0.54	1.20	1.32
Alumina and Ferric oxide	10.94	10.86	10.90
Lime	61.60	62.05	61.68
Magnesia	1.23	1.12	1.12
Sulphuric anhydride	1.13	1.48	1.37
Carbonic anhydride + Water	2.30	0.60	0.64
Alkalies and Loss	0.50	1.05	1.05
Total	100.00	100.00	100.00
Specific Gravity	3.135	3.195	3.185

[1]) The doubtful and the unsound samples were specially manufactured; they do not represent the normal output of the works, which habitually produces sound cement of high quality.

Mechanical Tests.

Tensile Strength: Kilos. per square cm.

	1 A. Sound.	2 A. Doubtful.	3 A. Unsound.
7 days neat	35.2	25.7	22.5
28 „ „	45.0	27.4	28.8
Gauged with	20% water	22% water	22% water
7 days sand (3:1)	16.2	10.9	9.8
28 „ „ (3:1)	21.8	17.8	15.5
Gauged with	9% water	9% water	9% water
Deval Test. 7 days neat at 80° C	42.0	Nil.	Nil.
Deval Test. 7 days sand at 80° C	22.2	Nil.	Nil.
Gauged with	9% water	9% water	9% water

Fineness.

Meshes per linear inch[1])	Residue on 50 mesh sieve	0.8%	0.8%	0.8%
	„ „ 76 „ „	3.2%	3.0%	3.6%
	„ „ 100 „ „	5.8%	6.0%	7.8%
	„ „ 180 „ „	17.4%	21.2%	24.8%
Setting Time (Vicat Needle)		3½ hrs	1 hr. 35 min.	7 mins: init. 25 „ final
Percentage of water for normal consistency		24%	26%	27·5%
Pat at 15° C for 28 days		Sound.	Sound.	Slightly buckled & cracked.
Pat at 50° C for 24 hours		Sound.	Slightly buckled & cracked	Much buckled & cracked
Pat at 100° C for 6 hours		Sound.	Disintegrated.	Disintegrated

It will be seen from these results that the 3 samples are

[1]) These values correspond approximately with 400, 900, 1550 and 5000 meshes per square cm.

similar in all respects except as regards soundness. They are well suited for use in this investigation.

The constancy of volume tests by 5 different observers are given on the sheet marked Table I.

On examining this table it is evident that the ordinary cold water pat test at 28 days is not capable of detecting with certainty a cement known to be unsound and that it fails completely to detect a cement of doubtful soundness.

It is also apparent that there is much difficulty in obtaining concordant results with the Bauschinger Apparatus used by different operators. Apart from possible errors of manipulation this difficulty may well be caused by slight movement of the small metal plates inserted at each end of the test piece, between which the measurement is made. If the cement is expansive a displacement of these plates is rendered more probable and the difficulty of obtaining concordant results will be increased.[1])

The Le Chatelier test, on the other hand, gives results which are fairly concordant for different operators. Such divergencies as exist may reasonably be attributed to the fact that the samples were not all tested at the same time, and however carefully they were packed and stored may have become slightly aerated in some cases and thus made less expansive. It is also clear that tests at 15° C and at 50° C do not suffice to detect a doubtful or unsound cement with certainty, but that when the test is carried out at 100° C as originally prescribed by Le Chatelier, it is easy to detect even a doubtful cement whereas a sound cement withstands the test perfectly, expanding to the extent of only a few millimetres.

The behaviour of the three samples referred to above is typical of that of the various other samples examined by different members of the Committee. In all, five sound samples, five doubtful

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[1]) The metal plates should be set in with cement known to be absolutely sound, not necessarily with the cement to be tested.

samples and four unsound samples were submitted to tests for constancy of volume. Without exception the cold pat test at 28 days was inadequate to detect with certainty doubtful samples or even samples known to be unsound. The tests at 50° C sufficed to detect samples thoroughly unsound but could not be relied on to detect doubtful samples. The only perfectly reliable tests were those at 100° C.

Several members of the Committee have expressed their views as to the adoption of a standard accelerated test for determining constancy of volume.

Mr. Leduc uses the Le Chatelier test at 100° C, habitually, allowing an expansion of 6 mm for marine construction and 12 mm for other classes of work.

Mr. Bienfait uses hot tests for all samples of cement passing through his hands; he considers the Bauschinger and Le Chatelier tests of great value and insists on the fact that cold tests alone are insufficient to guarantee freedom from expansion. Mr. Styles when acting as Chief Chemist for the Associated Portland Cement Manufacturers Ltd. constantly employed hot tests of various kinds. In the light of the present investigation he unhesitatingly recommends the adoption of the Le Chatelier method as an accelerated test for determining the constancy of volume of cement. He considers the cold pat test at 28 days to be of little use and the pat test at 50° C not to be relied upon in the case of doubtful cements.

Mr. Schwarz while considering that a hot test may not be requisite for cement to be used under water recognises its necessity for cement exposed to the air. He is of opinion that the test at 100° C is more convenient than that at 50° C and that although the Bauschinger test has the advantage of giving absolute measurements yet the Le Chatelier test is the simplest and most convenient in application.

Mr. Blount is of opinion that for the ordinary work of cement testing, apart from matters of research, the Le Chatelier method is

the simplest and most reliable form of accelerated test for constancy of volume. He has adopted it in his practice.

From the foregoing facts it appears that the Le Chatelier method may be prescribed as a standard accelerated test for the constancy of volume of cement.

76—78 York-Street.
Westminster,
London, S. W.

For the Committee:

Bertram Blount.

Tests made by 5 members of the

	Bauschinger tests. (Unit = 1/100 mm.)					
	In water 28 days	In water 7 days, elongation per cent				
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
Mr. E. Leduc		**In water 6 hours only.**			**In water 6 hours only.**	
1 A (Sound) . .	6.5 14	2 3.5 1.5	6 3.5 16	0.06 0.14	0.02 0.035 0.015	0.06. 0.03 0.16
2 A (Doubtful) .	37 13 43	17 27 7	Disintegrated.	0.37 0.13 0.43	0.19 0.27 0.07	Disintegrated.
3 A (Unsound) .	37 34 28	17 38 35	Disintegrated.	0.37 0.34 0.28	0.17 0.38 0.35	Disintegrated.
Mr. L. Bienfait		**In water 7 Days.**			**In water 7 Days.**	
1 A (Sound) . .	11	4	8	0.11	0.04	0.08
2 A (Doubtful) .	29	Bar broken 469	Disintegrated.	0.29	4.69	Disintegrated.
3 A (Unsound) .	42.5	267	Disintegrated.	0.42	2.67	Disintegrated.
Mr. R. C. Styles						
1 A (Sound) . .	2	2.5	8	0.02	0.025	0.08
2 A (Doubtful) .	8	40.0	1000 (Approx.)	0.08	0.40	10 (Approx.)
3 A (Unsound) .	10	109.5	1200 (Approx.)	0.10	1.09	12 (Approx.)

Committee on the constancy of volume. Table I.

Le Chatelier tests. (Divergence of Needles in mm.)			Pat Test		
In water 28 days.	In water 24 hours.	In water 6 hours.	For 28 days.	For 24 hours.	For 6 hours.
15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
	In water 6 hours only.				
Nil. 1mm. Nil.	Nil. 1mm. Nil.	2 3 2 / 2·3	Sound	Sound	Sound
3 4 4 / 3.7	5 6 6 / 6	50 49 52 / 50	Sound	Sound	Cracked
3 4 5 / 4	5 8 8 / 7	51 50 54 / 52	Sound	Sound	Cracked
Nil.	Nil.	Nil.	Sound		
Nil.	7	28 & 29 / 28·5	Sound		
1	5 7 / 6	37 35 / 36	Buckled & Cracked.		
Nil.	Nil.	1. 1.	Sound	Sound	Sound
1. 1	8 7 / 7.5	53 50 / 51.5	Sound	Slightly Buckled & Cracked.	Disintegrated
3. 3	14 13 / 13.5	56 54 / 55	Sligthly Buckled & Cracked.	Much Buckled and Cracked.	Disintegrated

	Bauschinger tests. (Unit = 1/100 mm.)					
	In water 28 days	In water 7 days, elongation per cent				
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
Mr. M. Petersen						
1 A (Sound) . .	No Bauschinger tests made.					
2 A (Doubtful) .						
3 A (Unsound) .						
Mr. E. Schwarz						
1 A (Sound) . .	5	3	2	0.05	0.03	0.02
2 A (Doubtful) .	17	33	616	0.17	0.33	6.16
3 A (Unsound) .	19	33	Over1000	0.19	0.33	Over 10.0

Table I.

Le Chatelier tests. (Divergence of Needles in mm.)			Pat Test		
In water 28 days.	In water 24 hours.	In water 6 hours.	For 28 days.	For 24 hours.	For 6 hours.
15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
0.5 1.0 / 0.75	1 0.5 / 0.75	2.5 1.5 / 2.0	Sound		
		Boiled for 1 hour only.			
3 2.5 / 2.75	6 5.5 / 5.75	39.5 38.5 / 39	Sound		
		Boiled for 1 hour only.			
6 5 / 5.5	9 9 / 9.0	43.0 43.5 / 43.25	Sound		
7 days only in water.					
0.75	0.50	1	Sound		
2.75	4.25	40.25	Swellings on surface.		
3	7.25	44.5	Swellings on surface.		

Appendix I.

Detailed programme for the use of the Committee.

The percentage of water necessary for obtaining normal consistency has been determined and will be notified to each member of the Committee who has undertaken to carry out the tests. If on account of changes from aeration or alteration due to local conditions the percentage of water is found unsuitable, each member of the Committee who encounters this diffiiculty is requested to determine the percentage of water for himself and to communicate the results to the Chairman. It is advisable to determine the "loss on ignition" over a bunsen burner for half an hour, also the specific gravity in order that any discrepancy in the results due to aeration may be explained.

In all cases the cement shall be gauged to normal consistency unless difficulty is experienced in preparing satisfactory test pieces with this percentage of water, in which case the operator must gauge the cement according to his best judgment recording the quantity used and notifying the Chairman. Good fresh water not too hard must be used for gauging; failing this distilled water should be used.

The tests for each sample of cement shall be as follows:

1. An ordinary pat say 10 c. m. long $\times$ 5 c. m. wide $\times$ 5 m. m. in thickness kept for 24 hours in moist air after gauging and then in water at 15° C for 28 days. The condition of the pat to be observed at the end of this time; afterwards from time to time up to the termination of the tests (say 1 year).
2. The Le Chatelier test to be carried out according to the instructions in Schedule A (appended herewith) at 15° C, at 50° C and at 100° C.
3. The Bauschinger test to be carried out according to the instructions in Schedule B (appended herewith) at 15° C, at 50° C and at 100° C.

Schedule A.

The Le Chatelier Tests.

(1) With immediate immersion in water.

Cement is gauged and filled into the mould on a plate of glass the ends of needles being held together. When the mould has been filled it is covered with a plate of glass held down by a small weight and the whole is immersed in water at 15° C for 24 hours. 3 sets of tests are made.

(a) The test piece is allowed to continue in water at 15° C for 28 days.

(b) The test piece after remaining in water at 15° C for 24 hours is raised to a temperature of 50° C in the course of half an hour and remains immersed in water at this temperature for 24 hours.

(c) The test piece after remaining in water at 15° C for 24 hours is raised to a temperature of 100° C in the course of half an hour and remains immersed in water at this temperature for 6 hours.

In all cases the divergence of the needle is measured in mm. after removal from the water and in the case of the hot test pieces measured after cooling.

(2) A similar set of tests may be made on test pieces kept in moist air for 24 hours and then immersed in water.

It will be observed that this series of tests involves the preparation of 6 testpieces for each sample of cement examined.

Schedule B.

The method for the Bauschinger test to be this: 2 bars, of normal consistency, to be prepared of each cement for each temperature of testing. One of these to be immersed immediately in water and measured after 24 hours. The other to be kept in moist air for 24 hours, measured and then immersed. Further measurements to be made 7 days after the time of immersion in the case of the bars tested at 15° C, and at 1, 2, 3, 4, 5, 6 and 7 days after immersion in the case of the bars tested at 50° C and 100° C. Measurements to be continued in all cases at intervals of 7 days until the length of the bars remains constant.

Appendix II.

Tests made by Mr. M. Petersen.

Marked	Specific gravity	Loss on ignition with bunsen burner	Le Chatelier: In water 28 days at 15° C. (1)	Le Chatelier: In water 28 days at 15° C. (2)	Le Chatelier: In water 24 hours at 50° C. (1)	Le Chatelier: In water 24 hours at 50° C. (2)	Le Chatelier: In water 6 hours at 100° C. (1)	Le Chatelier: In water 6 hours at 100° C. (2)	Pats: Cold water for 6 months	% of water used: Sand (3:1)	% of water used: Neat	7 Days: Mortar (3:1): Tensile strength kilogr. per sq. cm.	7 Days: Mortar (3:1): Compression strength kilogr. per sq. cm.	7 Days: Neat Cement: Tensile strength kilogr. per sq. cm.	7 Days: Neat Cement: Compression strength kilogr. per sq. cm	28 Days: Mortar (3:1): Tensile strength kilogr. per sq. cm.	28 Days: Mortar (3:1): Compression strength kilogr. per sq. cm.	28 Days: Neat Cement: Tensile strength kilo per sq. cm.	28 Days: Neat Cement: Compression strength kilogr. per sq. cm.	Erdmenger Test[2]) Test Pieces 3:1: Tensile strength kilogr. per sq. cm.	Erdmenger Test[2]) Test Pieces 3:1: Compression strength kilogr. per sq. cm.
1 A	3.10	2.50%	0.5	1.0	1.0	0.5	2.5	1.5	Sound	8	18½	15.6	144.4	36.4	Not made	22.3	208.2	47.4	504.0	29.5	335.8
2 A	3.14	0.45%	3.0	2.5	6.0	5.5	39.5[1]	38.5[1]	Sound	8½	22	11.3	126.8	30.6	„	17.8	188.6	34.9	289·8	Cracked and swollen and had no strength that could be measured	
3 A	3.15	0.60%	6.0	5.0	9.0	9.0	43.0[1]	43.5[1]	Sound	9	23	7.8	93.2	26.2	„	16.8	151.2	33.2	235.8		

[1]) Only heated for 1 hour. Test pieces showed cracks.

[2]) Test pieces kept in moist air 24 hours and then 2 days in water. Afterwards heated in the Erdmenger boiler 6 hours at a pressure of 10 atmospheres.

	1 A. sound	2 A doubtful	3 A unsound	1 B sound	2 B doubtful	I sound	II sound	III sound	1 C	1 E sound	2 E doubtful	3 E unsound	II' doubtful	III' unsound
	Per cent													
Silica	21.76	21.64	21.92	22.02	22.40	25.06	23.84	23.08	23.60	21.52	21.64	20.62	26.12	24.10
Insoluble residue	0.54	1.20	1.32	0.74	2.14	1.54	1.52	1.26	1.14	0.92	1.00	1.20	0.64	0.70
Alumina and Ferric oxide	10.94	10.86	10.90	10.70	10.00	5.06	5.04	5.04	8.16	10.40	10.26	9.80	4.66	4.74
Lime	61.60	62.05	61.68	61.66	61.32	63.32	65.16	66.12	62.90	61.80	62.10	63.20	66.44	67.64
Magnesia	1.23	1.12	1.12	1.26	1.23	0.76	0.79	0.79	1.04	1.08	1.04	1.01	0.54	0.60
Sulphuric anhydride	1.13	1.48	1.37	0.79	1.27	1.13	0.62	0.89	1.37	1.16	1.13	1.20	0.20	0.21
Carbonic anhydride and water	2.30	0.60	0.64	2.04	0.80	2.56	2.56	2.40	1.08	2.16	1.96	2.26	0.94	1.20
Alkalies and loss	0.50	1.05	1.05	0.79	0.84	0.57	0.47	0.42	0.71	0.96	0.87	0.71	0.68	0.81
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Specific gravity	3.135	3.195	3.185	3.185	3.155	3.086	3.095	3.077	3.155	3.105	3.125	3.125	3.145	3.145

Cement No. I A. **Tests made by**

Normal Consistency	Loss on ignition on bunsen burner	Specific Gravity	Pat 5 mm. thickness	Le Chatelier Tests Immersed immediately in water at 15° C.		
				28 days in water at 15° C.	24 hours in water at 50° C.	6 hours in water at 100° C.
Setting begins after 1 hour 33 minutes Time of setting 2 hours 36 minutes Temperature 17° C. Gauged with 24% water Rise of Temperature 0° C.	1.68%	3,14	after 28 days normal	No divergence of needles	No divergence of needles	No divergence of needles

Analysis of Cement I A.

Insoluble matter	0.42%
Silica (SiO_2)	21.59%
Oxide of iron & alumina	10.10%
Calcium oxide	62.76%
Magnesium oxide	1.17%
Sulph. Acid (SO_3)	1.35%
Alkali	0.38%
Water	0.54%
Loss on ignition	1.68%
	99·99%

Bauschinger Tests

Elongations in 1/200 mm.

Six hours in moist air, remains in water 15° C.	Kept in moist air at 15° C.	Kept in water at 50° C. 7 days	Kept in water at 100° C. 7 days
Sept. 24 th. = a + 5,16	Sept. 24 th. = a + 5,98	Sept. 29 th. = a + 5,73	Oct. 10 th. = a + 6,36
Oct. 1 st. = a + 5,33	Oct. 1 st. = a + 5,92	Sept. 30 th. = a + 5,77	Oct. 11 th. = a + 6,42
Oct. 8 th. = a + 5,37	Oct. 8 th. = a + 5,87	Oct. 1 st. = a + 5,77	Oct. 12 th. = a + 6,48
Oct. 18 th. = a + 5,40	Oct. 15 th. = a + 5,85	Oct. 2 nd. = a + 5,79	Oct. 13 th. = a + 6,48
Oct. 22. nd. = a + 5,38	Oct. 22 nd. = a + 5,80	Oct. 3 rd. = a + 5,79	Oct. 14 th. = a + 6,51
Oct. 29 th. = a + 5,38	Oct. 29 th. = a + 5,79	Oct. 4 th. = a + 5,80	Oct. 15 th. = a + 6,52
	Nov. 5 th. = a + 5,77	Oct. 5 th. = a + 5,80	Oct. 16 th. = a + 6,52
	Nov. 12 th. a + 5,74	Oct. 6 th. = a + 5,81	
	Nov. 19 th. = a + 5,71	Oct. 7 th. = a + 5,81	
	Nov. 26 th. = a + 5,69		
	Dec. 3 rd. = a + 5,65		
	goes on steadily shrinking		

Cement Nr. 2 A.

Normal Consistency	Loss on ignition on bunsen burner	Specific Gravity	Pat 5 mm thicknes.	Le Chatelier tests Immersed immediately in water at 15° C.		
				28 days in water at 15° C.	24 hours in water at 50° C.	6 hours in water at 100° C.
Initial set 12 minutes Final set 34 minutes Rise of Temperature 5° C. Gauged with 26% water	1,38	3,125	after 28 days normal.	No divergence of needles	Divergence of needles after 24 hours 7 mm. 24 hours more, same	Divergence of needles after 6 hrs. No. 1 = 29 mm. No 2 = 28 mm.

Analysis of Cement No 2 A.

Insoluble matter	1.20%
Silica (SiO_2)	21.84%
Oxide of iron & alumina	8.58%
Calcium oxide	63.04%
Magnesium oxide	1.33%
Sulph. Acid. (SO_3)	1.72%
Alkali	0.59%
Water	0.25%
Loss on ignition	1.38%
	99.93%

Bauschinger tests

Elongation in 1/200 mm.

Six hours in moist air remains in water 15° C.	Kept in moist air at 15° C.	Kept in water at 50° C. 7 days	Kept in water at 100° C. 7 days
Shows fissures after 2 or 3 days in water	Takes curved form after 2 or 3 days	Shows very soon heavy fissures, takes curved form, falls into 2 parts after 7 days	Shows immediately large deformation, heavy fissures, falls into 2 parts after 12 hours in water; these parts fall to pieces by taking them out of the bath.
Sept. 24 th. = a + 7,27	Sept. 24 th. = a + 7,31		
Oct. 1 st. = a + 7,66	Oct. 1 st. = a + 7,44	Sept. 29 th. = a + 9.44	
Oct. 8 th. = a + 7,75	Oct. 8 th. = a + 7,40	Sept. 30 th. = a + 9,67	
Oct. 15 th. = a + 7,84	Oct. 15 th = a + 7,38	Oct. 1 st. = a + 11,88	
Oct. 22 nd. = a + 7.85	Oct. 22 nd. = a + 7,32	Oct. 2 nd. = a + 17,63	
Oct. 29 th. = a + 7,85	Oct. 29 th. = a + 7,31	Oct. 3 rd. = a + 18,83	
	Nov. 5 th. = a + 7,29	Oct. 4 th. = a + 18,87	
	Nov. 12 th. = a + 7,28	Oct. 5 th. falls into 2 parts	
	Nov. 19 th. = a + 7,24		
	Nov. 26 2th. a + 7,2		
	Dec. 3 rd. a + 7,18 =		
	goes on steadily shrinking		

Cement Nr. 3 A.

Normal Consistency	Loss on ignition on bunsen burner	Specific Gravity	Pat 5 mm. thickness	Le Chatelier tests Immersed immediately in water at 15° C.		
				28 days in water at 15° C.	24 hours in water at 50° C.	6 hours in water at 100° C.
Initial set $5^1/_2$ minutes Final set $10^1/_2$ minutes Rise of Temperature 70° C.	2,25	3,125	After 24 hours in water the pat loosened from the glass plate, showed heavy fissures	Divergence of needles 1 mm.	Divergence of needles after 24 hours No. 1 = 5 mm. No. 2 = 7 mm. 24 hours more, no difference	Divergence of needles after 6 hrs. No. 1 21—56 = 35 mm. No. 2 17—54 = 37 mm.
Gauged with water $27^1/_2$ %					Shows fissures after test	Shows fissures after test

Analysis of Cement No. 3 A.

Insoluble matter	= 0,89%
Silica (SiO_2)	= 21,79%
Oxide of iron & alumina	= 10,22%
Calcium oxide	= 61,08%
Magnesium oxide	= 1,42%
Sulph. Acid (SO_3)	= 1,42%
Alkali	= 0,48%
Water	= 0,40%
Loss on ignition	= 2,25%
	99,95%

Bauschinger tests

Elongations in 1/200 mm.

Six hours in moist air remains in water 15° C.	Kept in moist air at 15° C.	Kept in water at 50° C. 7 days	Kept in water at 100° C. 7 days
Shows after 7 days, fissures and takes curved form	Shows after 2 days, fissures and curved form	Shows immediately heavy fissures and curved form	Takes immediately large fissures, falls to powder and pieces after 12 hours in water
Sept. 24th. = a + 6,99	Sept. 24th. = a + 7,29	Sept. 29th. = a + 6,34	
Oct. 1st. = a + 7,69	Oct. 1st. = a + 7,92	Sept. 30th. = a + 10,57	
Oct. 8th. = a + 7,81	Oct. 8th. = a + 7,88	Oct. 1st. = a + 11.44	
Oct. 15th. = a + 7,84	Oct. 15th = a + 7,87	Oct. 2nd. = a + 11,64	
Oct. 22nd. = a + 7,84	Oct. 22nd. = a + 7,82	Oct. 3rd. = a + 11,68	
Oct. 29th. = a + 7,84	Oct. 29th. = a + 7,80	Oct. 4th. = a + 11,69	
	Nov. 5th. = a + 7,78	Oct. 5th. = a + 11,73	
	Nov. 12th. = a + 7,75	Oct. 6th. = a + 11,73	
	Nov. 19th. = a + 7,73	Oct. 7th. = a + 11.73	
	Nov. 26th. = a + 7,71		
	Dec. 3rd. = a + 7,67		
	goes on steadily shrinking		

Zement I.

Analyse: SiO_2 = 26,85%
$Fe_2O_3 + Al_2O_3$ = 5,48%
CaO = 65,74%
MgO = 0,84%
SO_3 = 1,09%
100,00%

Glühverlust = 3,15%
Spezifisches Gewicht = 3,07
Bindezeit bei 24° C.: Beginn nach 3 St. — Min.
Schluß „ 6 „ 30 „
Wassermenge = 29,5%
Rückstand auf dem 900 cm^2 Siebe: 6%
„ „ „ 4900 cm^2 „ 18%

Bauschingerproben
Ausdehnung eines 100 *mm* langen Stabes in 1/100 *mm*

Nach Tagen	Unter Wasser abgebunden			An der Luft abgebunden		
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
1	—	2	5	—	2	4
2	—	2	5	—	3	4
3	—	3	5	—	3	4
4	—	4	5	—	3	5
5	—	4	5	—	3	5
6	—	4	6	—	4	5
7	2	4	5	1	4	6
14	3	4	5	1	4	6
21	3	4	6	3	5	5
28	2	4	6	3	5	6
35	3	4	6	2	6	6
42	3	5	6	1	5	6
49	3	5	6	3	5	6
56	3	4	6	1	5	6
63	3	4	6	1	6	5
70	2	5	6	1	6	7
77	2	5	6	1	5	7
84	2	5	6	1	6	6
91	2	5	8	1	5	6
98	2	5	6	1	6	7
105	2	5	7	1	6	7
112	2	5	8	1	6	7
119	2	5	8	1	6	8
126	2	5	8	1	6	8
133	2	5	8	1	5	8
140	2	4	7	1	6	8
147	2	5	7	1	6	8
154	2	5	8	1	6	8

Zement II.

Analyse SiO_2 = 26,21%
$Fe_2O_3 + Al_2O_3$ = 5,23%
CaO = 67,34%
MgO = 0,74%
SO_3 = 0,48%
100,00%

Glühverlust = 1,61%
Spezifisches Gewicht = 3,11
Bindezeit bei 24.5° C.: Beginn nach 0 St. 2 Min.
Schluß „ 4 „ — „
Wassermenge = 30%
Rückstand auf dem 900 cm^2 Siebe: 1%
„ „ „ 4900 cm^2 „ 14%

Bauschingerproben
Ausdehnung eines 100 mm langen Stabes in 1/100 mm

Nach Tagen	Unter Wasser abgebunden			An der Luft abgebunden		
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
1	—	18	248 [1]	—	24	192 [1]
2	—	19	249	—	25	194
3	—	21	249	—	26	195
4	—	21	250	—	26	196
5	—	22	250	—	26	196
6	—	21	250	—	26	196
7	9	21	250	8	25	195
14	10	23	250	9	27	196
21	11	23	251	10	28 [2]	195
28	11	23	251	10	28	196
35	11	23	252	11	28	196
42	11	23	251	11	28	197
49	11	22	252	11	28	196
56	13	22	252	11	28	196
63	12	23	252	11	28	197
70	11	23	251	11	27	196
77	12	23	252	11	28	196
84	12	24	252	11	28	197
91	11	23	251	11	28	197
98	12	23	252	11	28	196
105	12	23	252	11	28	197
112	12	23	252	10	27	197
119	12	23	252	11	27	197
126	12	23	252	11	27	197
133	12	23	252	11	27	198
140	12	24	252	11	27	198
147	12	24	252	11	27	198
154	12	24	252	11	27	197

[1]) Rissig. [2]) Schwach netzrissig.

Zement III.

Analyse: SiO_2 = 25,27%
$Fe_2O_3 + Al_2O_3$ = 5,08%
CaO = 68,49%
MgO = 0,56%
SO_3 = 0,60%
100,00%

Glühverlust = 2,01%
Spezifisches Gewicht = 3,10
Bindezeit bei 24° C.: Beginn nach 1 St. 30 Min.
Schluß „ 3 „ 30 „
Wassermenge: 27,5%
Rückstand auf dem 900 cm^2 Siebe: 1%
„ „ „ 4900 „ „ 12%

Bauschingerproben.
Ausdehnung eines 100 *mm* langen Stabes in 1/100 *mm*.

Nach Tagen	Unter Wasser abgebunden			An der Luft abgebunden		
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
1	—	189	Über	—	227	Über
2	—	191	1000[1]	—	230	1000[1]
3	—	191		—	231	
4	—	191		—	231	
5	—	192		—	231	
6	—	192		—	231	
7	20	192		15	231	
14	21	193		17	232	
21	22	193		18	232	
28	23	194		19	233	
35	23	194		19	233	
42	24	194		20	233	
49	25	194		21	233[2]	
56	24	194		21	233	
63	24	194		21	233	
70	25	193		20	233	
77	25	193		21	233	
84	25	193		20	233	
91	24	193		21	233	
98	25	193		21	233	
105	25	193		21	232	
112	25	193		21	233	
119	25	193		21	233	
126	25	193		21	233	
133	25	193		21	232	
140	25	193		21	232	
147	25	193		21	232	
154	25	193		22	233	

[1]) Starke Risse. [2]) Schwach netzrissig.

Zement II¹.

Analyse: SiO_2 = 26,58%
Fe_2O_3 + Al_2O_3 = 5,45%
CaO = 67,30%
MgO = 0,34%
SO_3 = 0,33%
100,00%

Glühverlust = 0,44%
Spezifisches Gewicht = 3,13
Bindezeit bei 27° C.: Beginn nach 0 St. 10 Min.
Schluß „ 5 „ 30 „
Wassermenge 31%
Rückstand auf dem 900 cm^2 Siebe: 0,5%
„ „ „ 4900 „ ² „ 4,5%

Bauschingerproben
Ausdehnung eines 100 *mm* langen Stabes in 1/100 *mm*.

Nach Tagen	Unter Wasser abgebunden			An der Luft abgebunden		
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
1	—	114	533[1])	—	75	499[1])
2	—	115	534	—	75	504
3	—	115	535	—	76	505
4	—	116	535	—	77	505
5	—	116	535	—	76	505
6	—	115	535	—	76	505
7	17	115	535	11	76	504
14	18	116	535	12	75	505
21	19	116	535	12	76	505
28	18	116	535	12	76	505
35	19	117	536	12	76	505
42	21	117	535	12	77	505
49	19	117	535	12	77	505
56	19	117	536	13	77	505
63	19	117	535	13	77	506
70	19	117	535	13	76	506
77	19	117	536	13	76	507
84	19	117	536	13	76	508
91	19	117	536	14	76	508
98	19	117	536	13	76	508
105	19	117	536	13	76	508
112	20	117	536	14	76	508
119						
126						
133						
140						
147						
154						

[1]) Risse.

Zement III'.

Analyse:

SiO_2	= 24,93%
$Fe_2O_3 + Al_2O_3$	= 5,00%
CaO	= 68,78%
MgO	= 0,68%
SO_3	= 0,36%
$K_2O + Na_2O +$ Diff.	= 0,25%
	100,00%

Glühverlust = 1,19%

Spezifisches Gewicht = 3,13

Bindezeit bei 27° C.: Beginn nach 0 St. 5 Min.
Schluß „ 5 „ — „

Wassermenge = 30,5%

Rückstand auf dem 900 cm^2 Siebe: 0,5%
„ „ „ 4900 cm^2 „ 5,0%

Bauschingerproben
Ausdehnung eines 100 *mm* langen Stabes in 1/100 *mm*

Nach Tagen	Unter Wasser abgebunden			An der Luft abgebunden		
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
1	—	117[1])	Über 800[2])	—	79[1])	Über 800[2])
2	—	118		—	80	
3	—	120		—	81	
4	—	119		—	81	
5	—	119		—	82	
6	—	120		—	82	
7	15	121		14	81	
14	16	120		13	82	
21	17	122		15	83	
28	18	122		14	84	
35	17	123		15	84	
42	19	123		14	84	
49	19	124		16	84	
56	17	123		14	84	
63	17	123		14	85	
70	17	122		14	84	
77	18	122		15	84	
84	18	123		16	84	
91	18	123		15	84	
98	17	122		16	84	
105	18	123		16	84	
112	18	123		16	84	

[1]) Haar rissig. [2]) Starke Risse.

Zement 1A.

Analyse:

SiO_2	= 23,27%
$Fe_2O_3 + Al_2O_3$	= 10,83%
CaO	= 63,16%
MgO	= 1,28%
SO_3	= 0,85%
$K_2O + Na_2O +$ Diff.	= 0,61%
	100,00%

Glühverlust = 2,53%

Spezifisches Gewicht = 3,09

Bindezeit bei 23° C.: Beginn nach 0 St. 25 Min.
Schluß „ 1 „ 45 „

Wassermenge = 24%

Rückstand auf dem 900 cm^2 Siebe: 3%
„ „ „ 4900 cm^2 „ 10%

Bauschingerproben
Ausdehnung eines 100 *mm* langen Stabes in 1/100 *mm*

Nach Tagen	Unter Wasser abgebunden			An der Luft abgebunden		
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
1	—	2	3	—	2	2
2	—	2	3	—	2	3
3	—	4	3	—	2	2
4	—	2	2	—	2	2
5	—	2	2	—	2	2
6	—	3	2	—	3	3
7	4	3	2	2	4	3
14	5	4	3	3	3	4
21	5	4	3	3	4	3
28	5	4	3	3	4	3
35	5	3	3	3	4	3
42	6	4	4	4	4	4
49	6	4	4	4	3	4
56	6	4	4	3	3	4
63	6	3	4	4	3	4
70	6	3	4	4	3	4

Zement 2A.

Analyse: SiO_2 = 23,56%
$Fe_2O_3 + Al_2O_3$ = 10,56%
CaO = 62,24%
MgO = 0,99%
SO_3 = 1,43%
$K_2O + Na_2O +$ Diff. = 1,22%
100,00%

Glühverlust = 1,27%
Spezifisches Gewicht = 3,15

Bindezeit bei 23° C.: Beginn 0 St. 2 Min.
Schluß 0 „ 4 „

Wassermenge: 26%

Rückstand auf dem 900/cm^2 Siebe: 3%
„ „ „ 4900/cm^2 „ 11%

Bauschingerproben
Ausdehnung eines 100 *mm* langen Stabes in 1/100 *mm*

Nach Tagen	Unter Wasser abgebunden			An der Luft abgebunden		
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
1	—	30	611[1]	—	41	Über 1000[2] —
2	—	32	613	—	52	
3	—	33	614	—	56[2]	
4	—	33	615	—	56	
5	—	32	615	—	56	
6	—	34	615	—	57	
7	12	33	616	8	57	
14	14	35	617	10	58	
21	16	34	?	12	58	
28	17	34		13	59	
35	17	34		14	59	
42	18	34		14	59	
49	18	35		15	59	
56	19	35		14	61	
63	19	35		15	59	
70	20	35		15	59	
77	19	34		15	60	
84	19	36		15	61	
91	20	35		15	61	
98	20	37		15	61	
105	20	35		16	60	
112	20	35		16	60	
119	20	35		16	60	
126	20	36		16	62	
133	20	35		16	62	
140	21	35		16	61	
147	21	35		16	62	
154	21	35		16	62	

[1]) Starke Risse. [2]) Schwache Risse.

Zement 3A.

Analyse: SiO_2 = 23,83%
$Fe_2O_3 + Al_2O_3$ = 10,56%
CaO = 62,11%
MgO = 1,12%
SO_3 = 1,26%
$K_2O + Na_2O$ — Diff. = 1,12%
100,00%

Glühverlust = 1,44%
Spezifisches Gewicht = 3,14

Bindezeit bei 23° C.: Beginn 0 St. 2 Min.
Schluß 0 „ 4 „

Wassermenge: 27,5%

Rückstand auf dem 900/cm^2 Siebe: 3 %
„ „ „ 4900/cm^2 „ 10 %

Bauschingerproben
Ausdehnung eines 100 *mm* langen Stabes in 1/100 *mm*

Nach Tagen	Unter Wasser abgebunden			An der Luft abgebunden		
	15° C.	50° C.	100° C.	15° C.	50° C.	100° C.
1	—	29	Über	—	92	Über
2	—	31	1000[2]	—	135[1]	1000[2]
3	—	32		—	141	
4	—	32		—	144	
5	—	33		—	145	
6	—	33		—	146	
7	17	33		10	146	
14	17	34		11	147	
21	18	35		12	147	
28	19	35		13	148	
35	20	34		13	148	
42	20	34		14	148	
49	21	35		14	148	
56	21	36		16	148	
63	21	35		14	148	
70	21	35		16	148	
77	21	35		15	148	
84	21	35		16	148	
91	21	34		14	147	
98	21	34		14	147	
105	21	35		15	148	
112	21	35		15	148	
119	22	35		15	148	
126	22	34		15	148	
133	22	35		15	148	
140	21	34		15	147	
147	22	34		15	147	
154	21	34		16	147	

[1]) Mit Rissen durchsetzt. [2]) Starke Risse.

Le Chatelierproben.

Zement I.

Zunahme des Nadelabstandes in Millimetern

24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel
0		0		0,5	
0	0	0	0	0,5	0,5
1 Woche in Wasser von 15° C.		24 Stunden in Wasser von 50° C.		6 Stunden in Wasser von 100° C.	
0,5		0,5		1,0	
0	0,25	0,5	0,5	1,0	1,0
Summa . . .	0,25	Summa . . .	0,5	Summa . . .	1,5

Zement II.

Zunahme des Nadelabstandes in Millimetern

24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel
20,0 [1])		23,0 [1])		22,5 [1])	
19,0 [1])	19,5	23,0 [1])	23,0	10,5 [1])	21,0
1 Woche in Wasser von 15° C.		24 Stunden in Wasser von 50° C.		6 Stunden in Wasser von 100° C.	
3,0		8,0		31,5	
3,0	3,0	8,0	8,0	29,5	30,5
Summa . . .	22,5	Summa . . .	31,0	Summa . . .	51,5

[1]) An der Spaltstelle der Form ein klaffender Riß.

Zement III.

Zunahme des Nadelabstandes in Millimetern

24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel
39,5 [1])		30,0 [1])		33,0 [1])	
42,5	41,0	36,0 [1])	33,0	40,0 [1])	36,5
1 Woche in Wasser von 15° C.		24 Stunden in Wasser von 50° C.		24 Stunden in Wasser von 100° C.	
10,0		21,5		55,0	
9,0	9,5	21,5	21,5	52,0	53,5
Summa . . .	50,5	Summa . . .	54,5	Summa . . .	90,0

[1]) An der Spaltstelle der Form ein klaffender Riß.

Zement II¹.

Zunahme des Nadelabstandes in Millimetern

24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel
10,5		10,0		9,0	
11,5	11,0	11,0	10,5	10,5	9,75
1 Woche in Wasser von 15° C.		24 Stunden in Wasser von 50° C.		6 Stunden in Wasser von 100° C.	
3,5		13,0		35,0	
4,5	4,0	13,0	13,0	34,5	34,75
Summa . . .	15,0	Summa . . .	23,5	Summa . . .	44,5

Zement III¹.

Zunahme des Nadelabstandes in Millimetern

24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel
34,0 [1])		31,0 [1])		30,5 [1])	
35,0 [1])	34,5	34,5 [4])	32,75	30,0 [1])	30,25
1 Woche in Wasser von 15° C.		24 Stunden in Wasser von 50° C		6 Stunden in Wasser von 100° C.	
5,0		16,0		36,5	
5,0	5,0	17,5	16,75	36,5	36,50
Summa . . .	39,5	Summa . . .	49,5	Summa . . .	66,75

[1]) Klaffender Riß an der Spaltstelle.

Zement I A.

Zunahme des Nadelabstandes in Millimetern

24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel
0		0,5		1,0	
1	0,5	1,0	0,75	1,0	1,0
1 Woche in Wasser von 15° C.		24 Stunden in Wasser von 50° C.		6 Stunden in Wasser von 100° C.	
1,0		0,5		1,0	
0,5	0,75	0,5	0,50	1,0	1,0
Summa . . .	1,25	Summa . . .	1,25	Summa . . .	2,0

Zement II A.

Zunahme des Nadelabstandes in Millimetern

24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel
1,0 1,0	1,0	1,5 0,5	1,0	0,5 1,0	0,75
1 Woche in Wasser von 15° C.		24 Stunden in Wasser von 50° C.		6 Stunden in Wasser von 100° C.	
2,5 3,0	2,75	4,5 4,0	4,25	43,5 [1] 37,0 [1]	40,25
Summa . . .	3,75	Summa . . .	5,25	Summa . . .	41,00

[1]) Starke Risse.

Zement III A.

Zunahme des Nadelabstandes in Millimetern

24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel	24 Stunden in Wasser von 15° C.	Mittel
1,5 0,5	1,0	1,0 1,5	1,25	1,0 2,0	1,5
1 Woche in Wasser von 15° C.		24 Stunden in Wasser von 50° C.		6 Stunden in Wasser von 100° C.	
3,0 3,0	3,0	7,0 7,5	7,25	45,0 [1] 44,0 [1]	44,5
Summa . . .	4,0	Summa . . .	8,50	Summa . . .	46,0

[1]) Starke Risse.

Der Befund der Kuchenproben (pats) von 10 *cm* Länge, 5 *cm* Breite und 5 *mm* Dicke war folgender:

Kuchen aus Zement I: vollständig intakt
" " " II: " "
" " " III: " "
" " " II¹: " "
" " " III¹: " "
" " " 1 A: " "
" " " 2 A: vereinzelte Wärzchen auf der Oberfläche
" " " 3 A: " " " " "

Nach Tagen	Le Chatelierprobe bei 100° C. Nadelweite in Millim.	Darrkuchenprobe
0	52	starke Risse
3	45	ein Riß am Rande
5	42	ein schwacher Riß am Rande
7	38	„ „ „ „ „
10	28	„ „ „ „ „
12	27	Gestanden
14	24	„
17	17	„
19	16	„
21	11	„
24	10	„
26	10	„

Bauschinger.

	Eau à 15° C.		Air humide à 15° C.			Eau à 50° C.			Eau 100° C.		
Longueur initial . .	11,18	12,55	7,25	7,86	5,79	11,56	11,20	10,77	11,47	10,49	10,79
do. après 1 semaine	11,23 diff. +0,05	12,61 +0,06	7,18 −0,07	7,62 −0,24	5,67 −0,12	—	—	—	—	—	—
do. do. 2 do.	11,25 diff. +0,07	12,65 +0,10	7,18 −0,07	7,56 −0,30	5,67 −0,12	Après 6 heures à 50°			Après 6 heures à 100°		
do. do. 3 do.	11,39 +0,21	12,71 +0,16	7,21 −0,04	7,87 −0,01	5,77 −0,02	11,60 +0,04	11,27 +0,07	10,80 +0,03	10,59 +0,12	10,56 +0,07	11,11 +0,32
do. do. 4 do.	11,31 +0,13	12,83 +0,28	7,19 −0,06	7,70 −0,16	5,89 −0,10	—	—	—	—	—	—

Le Chatelier.

1 semaine	0	1	0	0	0	0	Après 6 heures à 50°			Après 6 heures à 100°		
2 do.	0	1	0	0	0	0						
3 do.	0	1	0	0	0	0						
4 do.	0	1	0	0	0	0	0	1	0	2	3	2

Galettes.

Aucune des galettes exposées à l'eau à 15° C., 50° C., 100° C. et à l'air humide n'a montré de fentes, gerçures ou fendillements.

	Eau à 15° C.		Air humide à 15° C.				Eau à 50° C.			Eau à 100° C.		
Longueur initiale .	10,15	9,89	13,00	10,04	9,99	12,96	14,02	10,76	11,04	12,24	11,20	11,81
do. après 1 semaine	10,00	10,06	13,12	10,30	10,20	13,13						
	− 0,15	+ 0,17	+ 0,12	+ 0,26	+ 0,21	+ 0,17						
do. do. 2 do.	10,03	10,16	13,32	10,30	10,17	13,14						
	− 0,03	+ 0,27	+ 0,32	+ 0,26	+ 0,18	+ 0,18	après 6 heures à 50° C.			après 6 heures à 100° C.		
do. do. 3 do.	10,64	10,18	13,17	10,07	10,14	13,33						
	+ 0,49	+ 0,29	+ 0,17	+ 0,03	+ 0,15	+ 0,37						
do. do. 4 do.	11,90	10,79	13,27	10,09	10,16	13,42	14,40	11,31	11,18	désagrégé		
	+ 0,75	+ 0,86	+ 0,27	+ 0,05	+ 0,17	+ 0,46	+ 0,38	+ 0,55	+ 0,14			

Le Chatelier.

Ecartement en millimetres après 1 semaine	3	4	4	2	2	2						
do. do. 2 do.	3	4	4	2	3	2	après 6 heures à 50° C.			après 6 heures à 100° C.		
do. do. 3 do.	3	4	4	2	3	2						
do. do. 4 do.	3	4	4	2	3	2	5	6	6	50	49	52
										Nombreux forts		

Galettes.

1 semaine			après 6 heures à 50° C.	après 6 heures à 100° C.
2 do.				
3 do.	Néant	Néant	Néant	Fentes
4 do.				

	Eau à 15° C.		Air humide à 15° C.				Eau à 50° C.			Eau à 100° C.		
Longueure initiale .	10,68	10,78	12,57	11.10	11,21	12,07	11,43	11,84	11,74			
do. après 1 semaine	11,13	10,92	12,15(?)	11,60	11,30	12,14						
	+ 0,45	+ 0,14	+ 0,42(?)	+ 0,50	+ 0,09	+ 0,07						
do. do. 2 do.	12,17	11,03	13,20	11,80	11,28	12,14						
	+ 0,49	+ 0,25	+ 0,63	+ 0,70	+ 0,07	+ 0,07	Après 6 heures à 50°.			Après 6 heures à 100°.		
do. do. 3 do.	12,37	11,31	13,10	11,82	11,31	12,16	11,79					
	+ 0,69	+ 0,53	+ 0,53	+ 0,72	+ 0,10	+ 0,09	+ 0,36					
do. do. 4 do.	12,43	11,43	13,13	11,81	11,27	11,14		12,61	12,43	Désagrégé.		
	+ 0,75	+ 0,65	+ 0,56	+ 0,71	+ 0,06	+ 0,07		+ 0,77	+ 0,69			

Le Chatelier.

Ecartement en millimetres												
après 1 semaine	1	2	2	0	0	0						
do. do. 2 do.	3	4	5	3	2	1	après 6 heures à 50°.			après 6 heures à 100°.		
do. do. 3 do.	3	4	5	3	2	1						
do. do. 4 do.	3	4	5	3	2	2	5	8	8	51	50	54
											Fentes	

Galettes.

1 semaine	Néant.	Néant.	Néant.	Fentes.
2 do.				
3 do.				
4 do.				

C. Styles

ater for hours fter aging	In 2- g
2.0	
3.5	
4.0	
4.0	
0.04%	
5.5	
3.5	
3.5	
3.5	
3.5	
3.5	
6.5	

OF THE

Results of tests made by Mr. R. C. Styles.

	1 A		2 A		3 A		1 B		2 B		1 E		2 E		3 E		II'		III'	
	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging	In air for 24 hours after gauging	In water for 24 hours after gauging
Bauschinger tests.																				
Elongation unit 1/100 mm.																				
15° After 7 days in water	2.0	2.0	4.5	4.5	5.5	6.5	0.5	0.5	1.5	2.0	2.0	2.0	8.5	8.5	15.5	45.0	24.5	44.5	25.5	46.5
15° „ 14 „ „ „	2.0	2.0	7.0	6.5	9.5	9.0	1.0	1.5	3.0	3.5	3.0	3.5	10.5	10.0	18.5	48.0	25.5	46.0	27.5	48.5
15° „ 21 „ „ „	2.0	2.0	7.5	7.5	10.0	9.5	2.0	2.5	3.5	4.0	3.5	4.0	11.0	11·0	19.5	48.0	25.5	46.0	27.5	48.5
15° „ 28 „ „ „	2.0	2.0	8.0	8.0	10.5	10.0	2.0	2.5	3.5	4.0	3.5	4.0	11.5	11.5	20.5	49.0	26.0	46.5	28.0	48.5
Elongation per cent	0.02%	0.02%	0.08%	0.08%	0.105%	0.10%	0.02%	0.025%	0.03%	0.04%	0.035%	0.40%	0.115%	0.115%	0.205%	0.49%	0.26%	0.46%	0.28%	0.48%
50° After 1 day in water	2.0	1.0	40.0	37.0	95.0	107.0	1.0	1.0	4.5	5.5	2.0	2.0	13.5	15.0	158.5	125.5	97.5	153.5	175.5	328.0
50° „ 2 days „ „	2.5	2.0	43.5	38.0	120.0	108.5	1.5	2.0	5.5	6.5	2.0	2.0	16.5	17.5	181.0	130.0	99.0	155.5	176.5	329.5
50° „ 3 „ „ „	3.0	2.0	45.5	38·5	125.0	109.5	2.0	2.5	7.0	6.5	2.0	2.0	17.5	18.0	184.5	134.5	99.5	155.5	177.5	330.5
50° „ 4 „ „ „	3.5	2.0	46.5	40.0	127.0	109.5	2.5	2.5	7.0	6.5	2.5	2.5	18.5	18.5	188.0	134.5	100.0	156.0	178.5	330.5
50° „ 5 „ „ „	3.5	2.5	46.5	40.0	127.5	109.5	2.5	2.5	7.0	6.5	3.0	2.5	19.5	19.0	189.0	134.5	100.0	156.0	178.5	330.5
50° „ 6 „ „ „	3.5	2.5	46.5	40.0	127.5	109.5	2.5	2.5	7.0	6.5	3.5	2.5	19.5	19.0	189.5	134.5	100.0	156.0	178.5	330.5
50° „ 7 „ „ „	3.5	2.5	46.5	40.0	127.5	109.5	3.0	3.0	7.0	6.5	3.5	3.0	19.5	19.0	189.5	134.5	100.0	156.0	178.5	330.5
Maximum elongation	3.5	2.5	46.5	40.0	3127.5	109.5	3.0	3.0	7.0	6.5	3.5	3.0	19.5	19.0	189.5	134.5	100.0	156.0	178.5	330.5
Elongation per cent	0.035%	0.025%	0.465%	0.40%	1.27%	1.09%	0.03%	0.03%	0.07%	0.065%	0.035%	0.08%	0.195%	0.19%	1.89%	1.34%	1.00%	1.56%	1.78%	3.30%
100° After 1 day in water	1.5	3.0	1000 (Approx)		1200 (Approx.)		5.0	5.5	35.5	14.0	3.5	3.5	225.5	198.0	1100		—	—	—	—
100° „ 2 days „ „	3.5	5.0	Bars too much swollen and cracked for accurate measurements				5.0	5.5	36.5	15.0	4.0	4.0	225.5	198.0	Bars too much swollen, and distorted for accurate measurements		—	—	—	—
100° „ 3 „ „ „	3.5	5.5	—	—	—	—	6.5	5.5	37.5	16.0	4.0	4.0	225.5	198.0	—	—	—	—	—	—
100° „ 4 „ „ „	4.5	6.0	—	—	—	—	6.5	5.5	37.5	16.0	4.0	4.0	225.5	198.0	—	—	—	—	—	—
100° „ 5 „ „ „	4.5	7.0	—	—	—	—	6.5	5.5	37.5	16.0	4.5	4.0	225.5	198.0	—	—	—	—	—	—
100° „ 6 „ „ „	5.0	8.0	—	—	—	—	6.5	5.5	37.5	16.0	5.0	4.5	225.5	198.0	—	—	—	—	—	—
100° „ 7 „ „ „	5.0	8.0	—	—	—	—	6.5	5.5	37.5	16.0	5.0	4.5	225.5	198.0	—	—	—	—	—	—
Maximum elongation	5.0	8.0	—	—	—	—	6.5	5.5	37.5	16.0	5.0	4.5	225.5	198.0	—	—	—	—	—	—
Elongation per cent	0.05%	0.08%	10.0% (Approx.)		12.0% (Approx.)		0.065%	0.055%	0.37%	0.16%	0.05%	0.045%	2.25%	1.98%	11% (Approx.)		—	—	—	—
Le Chatelier tests.																				
15° 28 days	Nil.	Nil.	1 mm.	1 mm.	3 mm.	3 mm.	Nil.	Nil.	Nil.	Nil.	Nil.	Nil.	—	—	—	—	—	—	—	—
50° 24 hours	Nil.	Nil.	8 mm.	7 mm.	14 mm.	13 mm.	1 mm.	1 mm.	2 mm.	2 mm.	Nil.	Nil.	2 mm.	2 mm.*	10 mm.	9 mm.	14 mm.	8 mm. cold + 17 mm. at 50° C. = 25 mm.	17 mm.	23 mm. cold + 22 mm. at 50° C. = 45 mm.
100° 6 hours	1 mm.	1 mm.	53 mm.	50 mm.	56 mm.	54 mm.	1 mm.	1 mm.	5 mm.	5 mm.	Nil.	Nil.	30 mm.	30 mm.	63 mm.	62 mm.	47 mm.	8 mm. cold + 41 mm. at 100° C. = 49 mm.	68 mm.	25 mm. cold + 48 mm. at 100° C. = 73 mm.
Normal consistency	24%		26%		27.5% (7 mins initial. 25 „ Final)		26%		24%	26%	27.5%		27%		27%		35%		36%	
Setting time (vicat needle)	3½ hours		1 hour 35 mins.				17½ hours		3 hours 20 mins.		25 Mins.		20 mins.		25 mins.		6 hours		4¼ hours	
Meshes per linear inch	50 76 100 180		50 76 100 180		50 76 100 180		50 76 100 180		50 76 100 180		50 76 100 180		50 76 100 180		50 76 100 180		50 76 100 180	
Residue on Sieves*) Per cent	0.8 3.2 5.8 17.4		0.8 3.0 6.0 21.2		0.8 3.6 7.8 24.8		0.2 0.4 1.0 11.2		0.6 5.4 10.8 27.2		Trace 0.6 2.0 13.0		0.2 1.7 7.0 23.3		0.9 4.3 11.3 34.5		Nil. 0 2 9.8 30.2		Nil. 0·3 11.0 33.1	
Pat at 15° for 28 days	Sound		Sound		Slightly buckled and cracked		Sound		Sound		Sound		Sound		Slightly buckled		Sound		Slightly buckled and cracked	
„ „ 50° „ 24 hours	Sound		Slightly buckled and cracked		Much buckled and cracked		Sound		Very slightly Buckled		Sound		Unsound		Disintegrated		Soft and much cracked		Disintegrated	
„ „ 100° „ 6 „	Sound		Disintegrated		Disintegrated		Sound													
Tensile tests (lbs. per sq. inch).																				
Neat 7 days cold	500		366		320		536		390		510		450		490		360		445	
Neat 7 „ deval	597	20% water	Nil.	22% water	Nil.	22% water	465	20% water	426	22% water	670	22% water	550	22% water	60	22% water	490 (a)	30% water	430 (a)	30% water
Neat 29 „ cold	640		390		410		716		560		570		615		600		450		525	
Sand 7 days cold	230		155		140		220		160		190		150		105		150		105	
Sand 7 „ deval	315	9% water	Nil.	9% water	Nil.	9% water	325	9% water	227	9% water	260	9% water	Nil.	9% water	Nil.	9% water	70 (a)	10% water	Nil.	10% water
Sand 28 „ cold	310		245		220		336		247		275		225		160		170		160	
Equivalent to KGMS per sq. CM.																				
Neat 7 days cold	35.2		25.7		22.5		37.7		27.4		35.6		31.7		34.5		25.3		31·3	
Neat 7 „ deval	42.0		Nil.		Nil.		32.7		30.0		47.1		38.7		4.2		34.5		30.3	
Neat 28 „ cold	45.0		27.4		28. 8		50.4		39.4		40.1		43.3		42.2		31.7		36.9	
Sand 7 days cold	16.2		10.9		9.8		15.5		11.3		13.3		10.6		7.4		10.6		7.4	
Sand 7 „ deval	22.2		Nil.		Nil.		22.9		16.0		18.3		Nil.		Nil.		4.9		Nil.	
Sand 28 „ cold	21.8		17.8		15.5		23.6		17.3		19.3		15.8		11.3		12.0		11.3	

17 c.

INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON THE PROBLEM 17.

Group C;

ON METHODS FOR TESTING PIPES.

PRESENTED BY PROFESSOR
M. GARY,
ABTEILUNGSVORSTEHER DES KÖNIGL.
MATERIALPRÜFUNGSAMTES IN GROSS-
LICHTERFELDE-W.

(TRANSLATED FROM THE GERMAN
ORIGINAL.)

UNIVERSITY OF

The testing of Pipes.

By

Professor M. Gary,

Abteilungsvorsteher des kgl. Materialprüfungsamtes in Groß-Lichterfelde-W.

(Translation of the German original.)

The Council of the International Association for Testing Materials has determined in the meeting, held the 1 and 2. Mai 1902, to dissolve the Committee hitherto in charge of the

Problem „Nr. 17 Introduction of uniform testing methods for pipes"

and to refer the settlement of this question to a single investigator.

The Council hopes that by this means the problem may be solved more quickly, and I have been asked by that body to report on this subject at the IVth Congress, originally intended te be held in St. Petersburg.

As far as the question of proposals regarding the introduction of certain testing-methods is concerned there is no essentially new material in hand, only a few new pieces of testing apparatus having been proposed. The chairman of Committee 17, nominated by the Council, was not in a position to discuss the solution of this problem in the absence of any co-operation; and had to confine himself to mentioning some literature on the question which had come to his knowledge through French periodicals.

I shall refer to this subject again later on.

At the International Conference for the standardisation of testing-methods, held in Munich Sept. 24 th 1884, Mr. Bergmann-Linz first expressed the desire to fix methods for testing the resistance of pipes to external and internal pressure. However

nothing was then done in regard to this suggestion. But at the request (Nov. 2th; 1896) of the German Stoneware factory, Friedrichsfeld Baden, the Council of the International Association has inserted in its technical programme the investigation of uniform methods of testing pipes.

On the suggestion of the Counicil I have, therefore, communicated with a number of experts in different countries and inserted the statements received, together with my own observations, in a report which I submitted to the Congress of the International Association held at Stockholm in the year 1897. At the same time I have raised the question of testing clay and cement pipes.

This report is published in the „Baumaterialienkunde" for 1897 (p. 360), and for 1898,) pp. 1, 17, 33 etc.) and I must refer to it here.

At that time I proposed for an exhaustive test of pipes the investigation of the following particulars :

1. Resistançe to outside pressure.
2. „ „ inside „
3. Structure of the material.
   a) Condition of the surface of fracture and nature of the cementing.
   b) Composition of the material (of cement pipes);
   c) Permeability (of cement pipes).
4. Condition of the inner surfaces :
   a) Resistance te acids (clay pipes);
   b) Wear (cement pipes).

Using the statements received at the time from different countries, I treated in detail each of the four points requiring investigation, and came to conclusions which I would now like to revise on consideration of more recent information.

In the meantime I may be allowed to give you a résumé of the French works on the subject to which the former chairman of Commission 17 referred as being remarkable.

Manufacture and Testing of Stone-ware Pipes.[1]).

[1]) Bulletin de la Société des ingénieurs et architectes sanitaires de France (1898—1900).

The unfortunate experiences in France following the use of stone-ware pipes for drainage purposes, especially after the year 1890, have reintroduced iron pipes, as being preferable.

For instance the defective stone-ware pipes in the Hospital Bouvicant were replaced by iron ones. The reason for this was however not in the main due to bad quality of the French stone-ware, but rather to lack of carefulness in laying the pipes, and sometimes too, as in the case mentioned, to disregard of local conditions. The land in which the drainage system in question was established, was frequently submerged by the river Seine and, besides stone-ware pipes were used here for the first time for conducting warm water. It is not, therefore, inexplicable, that the cement-joints should suffer. As the question of a sanitary, and at the same time suitable drainage is of public interest, and as the existence of the French stone-ware industry was endangered, a Commission was then formed for the object of removing as far as might be possible the prevailing inconveniences.

After much consideration, experimenting and observation this Commission came to the following conclusions:

1. The cement[1]) (slow setting or quick setting, natural or artificial) must always be purchased from those manufacturers who guarantee a good and uniform product under all circumstances.

This condition, being itself understood and adhered to by the Authorities, offers to contractors for drainage-work the absolutely necessary guarantee of constancy of volume.

2. Stone-ware pipes for drainage must have a solid impermeable texture with conchoidal fracture (as with porcelain) the necessary hardness and a definite "ring". Besides they must withstand the action of acids (hydro-fluoric acid excepted).

The salt-glazing should not be a so-called coating-glaze on the body but must form an integral part of the mass itself. The glazed or vitreous fragments must not adhere to the tongue, as with broken clay-pottery, should not be scratched by steel and not absorb more water than 2% of the total weight.

The resistance to inside pressure, measured on a pipe closed by two strong iron plates, and having a diameter up to 0·2 m, should not be less than 5 kg per sq. cm.

[1]) To be used for socket-joints.

3. The individual pipes must be cylindrical in shape and the outside surfaces must be exactly parallel to the pipe axis.

The cement joint between spigot and socket must be 6 cm in length. (fig. 1.)

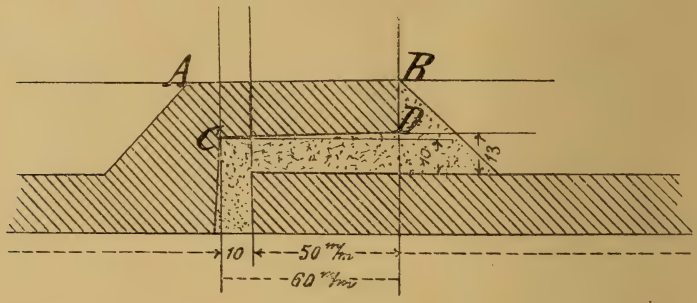

Fig. 1.

It is also advisable to have a slanting socket (C. D. not parallel to A. B.).

As can also be seen from the figure the connection to the following pipe must be concentric as it ensures a greater durability of the piping, and not as shown in figs. 2 and 3.

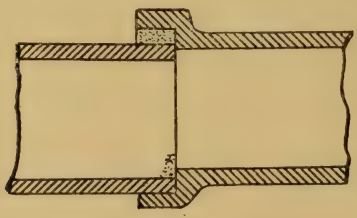

Fig. 2.

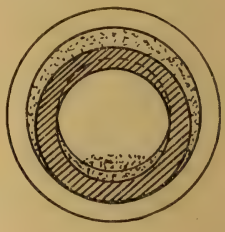

Fig. 3.

4. The pipes must bear the trademark of the factory and the designation of the quality (I, II or III), which means:

Quality I are perfectly burned and glazed pipes first class in every respect.

Quality II: pipes with insignificant flaws on the ends.

Quality III: good pipes but irregularly burned or glazed.

All others are best destroyed at once after their manufacture.

There are numerous remarks made in connection with these statements.

To 1. The following questions are raised by the consideration of the reasons why Portland cement is more suitable for the joints of piping than Roman cement; the causes which may give rise to warping and what means can be applied to accelerate the setting of the cement. And finally mention is made of the detrimental action certain ingredients of sewage water may exercise on the cement and the remedies for this.

The remarks on the manufacture refer to the ideal texture of the material as also to the kind and properties of the usual glazing and are of no importance. The remarks on

the testing of pipes

are of interest

a) to find the resistance to outside pressure, the pipes are tested by means of a double-armed lever. The pipe to be tested is placed under the edge of the lever and the latter weighted on both ends, until the pipe shows a complete fracture.

Repeated trials made with pipes from the different factories showed, in the case of good material, an average resistance of about 1500 kg, the variation as a rule being between the limits 1700 and 1200 kg.

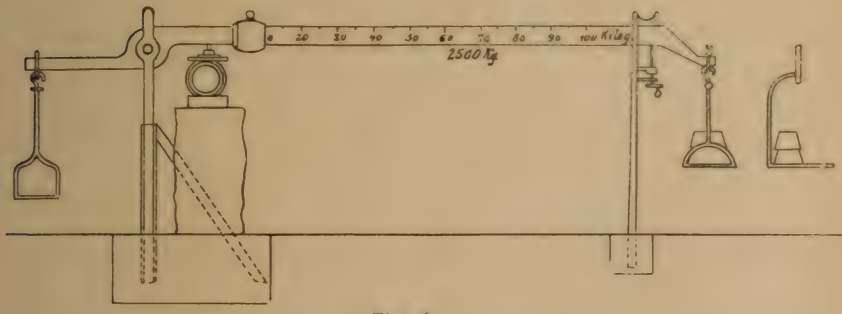

Fig. 4.

b) The strength of pipes when subjected to concussion is ascertained as follows: a steel ball of 6,5 kg weight is arranged to fall on the pipe under test. The ball is hung up by a thread to be burned through at a certain place. The test is repeated again and again, changing he length of drop,

until the pipe shows a crack. Generally the pipes withstand a blow from the ball from a height of 0·05, 0·10 and 0·15 m, some even from a height of 0·20 and 0·25 m.

c) The capacity for absorbing water is ascertained by comparing the weight of the dry pipe with its weight of that being submerged for 24 hours. The tests made in the laboratory show the average absorption to lie between the limits 14% and 28% (21%). It is rather more than the 15% mentioned in the official reports of the town of Paris, since material too well glazed and too fragile absorbs proportionally little water.

d) In order to test the resistance of pipes to internal pressure, a conduit of 4 to 5 meters in length is laid, the ends of the pipes closed, the pipes filled with water, the air being carefully removed and a pressure pump is operated until the fracture occurs. Numerous tests with French pipes have given good results. The average pressure was about 3·7 kg/sq. cm, the lowest being 2·0 to 2·5 kg, a result which may be considered as perfectly satisfactory (according to L. M. Masson).

As however the methods of testing under a) to d) can only be applied to certain individual pipes under the supervision of experts, and as furthermore each pipe of the conduit must be free from defects, it is of great importance to remind the workmen to test each pipe by knocking them before laying them. As a doctor convinces himself of the health of human beings by applying a stethoscope, each workman will, after some practice, be in a position to distinguish good pipes by their sound.

In respect of the methods for testing d) the Bulletin de la Société des ingénieurs et architectes sanitaires de France states further:

The resistance to internal pressure is no sure sign of the quality of a pipe and much less so if, as proposed by Masson the test is undertaken with a conduit of 4 to 5 meters in length, jointed with cement. Tests have proved conclusively that stoneware pipes alone stand even 19 atm. pressure, whereas the cement-joints of a conduit, built of the same pipes, allowed water to trickle through long before the pipes broke. For this reason it is advisable to use in the test a single pipe. This is filled with water,

placed horizontally between two plates and, after expulsion of the air, submitted to the water-pressure by means of a pressure pump. As in many instances material which is just porous allows water to trickle through long before the breakage but still withstands a proportionally high pressure, the observer must note whether water permeates the pipe before it fractures.

When testing the resistance to internal pressure, the diameter and thickness is to be considered also. According to tests made by Lencauchez stone-ware pipes stand a pressure which varies inversely as the diameter of the pipe. Besides the results given by the Commission refer only to drainage-pipes of a certain diameter (vis 0,20 m). Considering the proportions between thickness (e), diameter (D) and inside pressure (p) then (as per Bulletin de la Societé) the strength R against the inside pressure for 1 qcm can be found by the following formula:

$$R = \frac{p\ D}{2\ e}$$

hence

$$p = \frac{2\ e\ R}{D}$$

Assuming for instance the pipes to be 15 cm in diameter and 17 mm in thickness and that they stand 6 atm. pressure; then would

$$R = \frac{p\ D}{2\ e} = \frac{6 \times 15}{3{,}4} = 26{\cdot}4 \text{ kg/sq. cm.}$$

Therefore in the case of pipes of the same material, 40 cm in diameter, which generally have a thickness of 30 mm, the pressure should equal

$$\frac{2\ e\ R}{D} = \frac{6 \times 26{,}4}{40} = 3{\cdot}96 \text{ kg/sq. cm.}$$

Assuming that pipes of 15 cm diameter withstand a pressure of only 5 atm., we can deduce certain proportionate results which are useful in practice. In round figures, they are as follows:

Diameter of pipe.	Inside pressure.
5 to 15 cm	5 atm.
15 „ 25 „	4 „
25 „ 35 „	3 „
35 „ 50 „	2 „

In connection with the report given briefly above the following observations may be made.

Notwithstanding the efforts of the Commission it yet remains to be decided, whether under all circumstances cement-joints alone are to be taken into consideration for drain-pipes. The yearly consumption of more than 100.000 tons of pipes in Germany, where other methods of jointing are also used, compared with the yearly consumption of France of not more than 25.000 tons, gives rise to reflection especially as Germany offers for the manufacture of pipes no better clay than France does. The jointing material (2 parts of tar, 1 part of asphalte with oakum) proposed and used by Engineer Lindley for the drainage of Elberfeld has proved to be exceptionally unaffected by the action of acids.

The Commission therefore determined to communicate with this prominent engineer, and also with other prominent German experts such as M. Hobrecht and Dr. Weyl, in the interests of public welfare and the threatened French stone-ware industry.

Such is the report of the French engineers.

A good many of their conclusions do not appear to be fully proved (E. G. § 1), others are inapplicable (E. G. § 2 last part), and again others go too far and would be if taken literally impossible te carry out, as for instance § 3. Their report shows however

> that the consumer demands the right of controlling the pipes and that he must be allowed to carry out this control effectively.

Considering the views expressed in the French report and what I reported myself for the Stockholm Congress I wish to state first of all and ask the Congress at St. Petersburg to assert,

> „that the standardisation of the test-methods for clay and cement pipes is a necessity".

Assuming that the Congress takes this as understood, I beg to report briefly on the different test-methods proposed, referring to my suggestions in the Stockholm report, as follows:

Proposals for uniform tests of earthenware and cement pipes.

1. External pressure test.

a) Vertical pressure.

Actual experience seems to show that it is advisable to apply vertical pressure only and not to lay pipes in earth or sand, nor to use saddles surrounding the larger part of the surface of

the upper or lower part of the pipe. In order to adjust inequalities it is recommended to place pipes with a flat sole on a stratum of gypsum or sand, distributed on the lower pressure-plate.

The unevenness of round pipes can be adjusted either by the use of mortar packing or by inserting, vertically to the pipe axis, flat wedges of soft wood.

Either steel yards or hydraulic presses preferably the latter, may be used as testing-machines.

b) Concussion tests.

In order to find out the susceptibility of pipes to concussion, the use of a free falling ball, as shown in Fig. 5, is recommended. Instead of the thread to be burned through a clip, Fig. 6, may be used to liberate the weight.

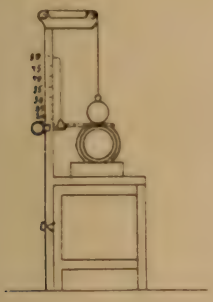

Fig. 5.

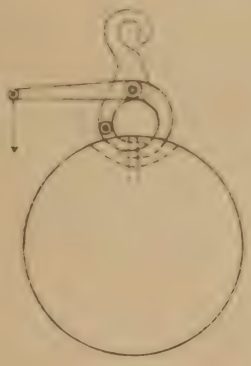

Fig. 6.

To obtain comparative figures it is necessary to find the force of the concussion required to shatter a sound pipe with one blow.

2. Test under internal pressure.

When testing single pipes under internal pressure they should be subjected only to forces acting radially. The insertion of the pipes between two rubber-jointed locking plates, which are pressed against the pipe ends in the direction of the axis, is to be rejected owing to the uncontrollable axial stresses involved.

Rudeloff recommends a more satisfactory arrangement[1]) (See Baumaterialienkunde 1898, page 17, Fig. 20). In this arrangement leather cups, filled up with gelatin, to secure a good fit, are placed in the pipe ends and are so fastened by cast iron or wooden cylinders, that it is impossible for them to slip out.

Into one of these cylinders the water inlet pipe packed by means of a leather cup is introduced. Two arrangements of this kind are illustrated below.

To calculate the strength T of the material from the inside pressure in atmospheres P the following formula:

$T = \frac{P\ d}{2\ W}$ serves as a basis, d being the interior diameter of pipe and W the thickness.

3. Examination of the texture.

a) Condition of the surface of fracture and kind of cementing.

The fracture of a good clay pipe must show thorough sintering and a solid structure without cracks. The material must be such that it can be worked with a chisel and hammer, and the pipe must, when hit with a hammer, give a metallic ring. The colour of the fracture taken by itself is insufficient evidence of the quality of clay-pipes, as the grey colouring of the fracture, which is often considered as a sign of thorough burning, can be also produced by steaming pipes made of porous material.

The fracture of cement pipes should also be homogeneous, free from cracks and uniform.

The pebbles in the cement-mortar shall be held so fast that they can be split by a hammer without their dropping out of the cement.

b) Permeability.

The permeability of pipes depends on the homogeneity of the surface and the porosity of the texture, or material of the pipe itself. The porosity of the body can easily be determined in the ordinary manner. The permeability of the inner and outer surfaces can be examined by placing on single fragments

[1]) See reports from the Königlich-technische Versuchsanstalten 1892, page 101.

of pipes graduated glass tubes of about 45mm inside diameter, adjusting the fragments horizontally and observing the time taken for water to penetrate the pipe.

The outer surfaces should be tested in the same manner as the inner surfaces, because where pipes have to be laid in water, the contents of the pipog should not come into contact with this water as would probably be the case where diffusion was possible.

4. Testing inner surfaces.

The testing of the inner surfaces of clay pipes is incomplete unless their resistance to the action of acid has been ascertained and, in the case of cement pipes, their resistance to wear.

For the testing of the surfaces or the glazing of the pipes in respect to their resistance to the action of acids, the method introduced by Kämmerer is recommended, which is as follows: Test pieces are exposed at the temperature of an ordinary room for 48 hours to te action of 1% solutions of hydrochloric acid, nitric acid, sulphuric acid and ammonia. Result may also be obtained by treating the glaze with vapours of fuming hydrochloric acid.

Changes in the weight and the physical condition of the test pieces are determined.

Seeger & Cramer's method is recommended for testing the material as regards its resistance to the action of acids. From the fragments to be tested, grains of distinct size are separated out by means of sieves having 121 and 64 meshes respectively to the square centimeter. The grains are washed with water to remove the dust, dried until the weight remains constant and then treated with ten times their volume of hydrochloric or nitric acid for a period of 24 hours, shaking them from time to time. The loss in weight is noted.

It is not necessary to test cement-pipes as regards their resistance to the action of acids, because, as is well known, cement-pipes withstand the action of very weak acids for long periods. But it is important to test the smoothness and resistance to friction of the inner coating of cement-pipes, on account of

the sand and other solid matter often contained in sewage. As owing to the shape of the pipe surfaces the usual attrition process cannot be adopted, I would recommend for testing the thickness and durability of the inner coating of cement-pipes a sand blast according to a method introduced by myself.

A test-piece, circular in form and 6 cm in diameter, is exposed for two minutes to a sand blast actuated by steam at 2 atm. pressure.

The details of the method and its results are published in the „Mitteilungen aus dem königl. Materialprüfungsamte" (1904, page 103—123).

On the evidence of the aforesaid statements and those already published I move that

»the Congress desires to recommend the following methods for testing clay and cement pipes:

1. The test of resistance to external strains by
   a) applying vertical pressure,
   b) using a drop-weight.
2. The test of resistance to uniform, radially acting internal pressure, eliminating axial tensions.
3. Testing the texture.
   a) By hammer blows.
   b) By determination of the porosity.
4. Testing the internal surfaces.
   a) For resistance of the glaze to acids.
   b) For resistance of the body of the ware to acids.
   c) Resistance of the internal skin of cement pipes to the sand blast.

For the performance of these tests Professor Gary's methods given in his report presented to the St. Petersburg Congress in 1904, shall be the standard methods.«

Testing apparatus.

In extension of the foregoing statements, I beg also to submit to the Congress the following testing apparatus introduced into Germany in the course of the last few years.

1. Presses for pipes.

a) The press of the German Beton-Verein (Fig. 7), designed by Koenen and built by Grether & Co. (Freiburg, Baden).

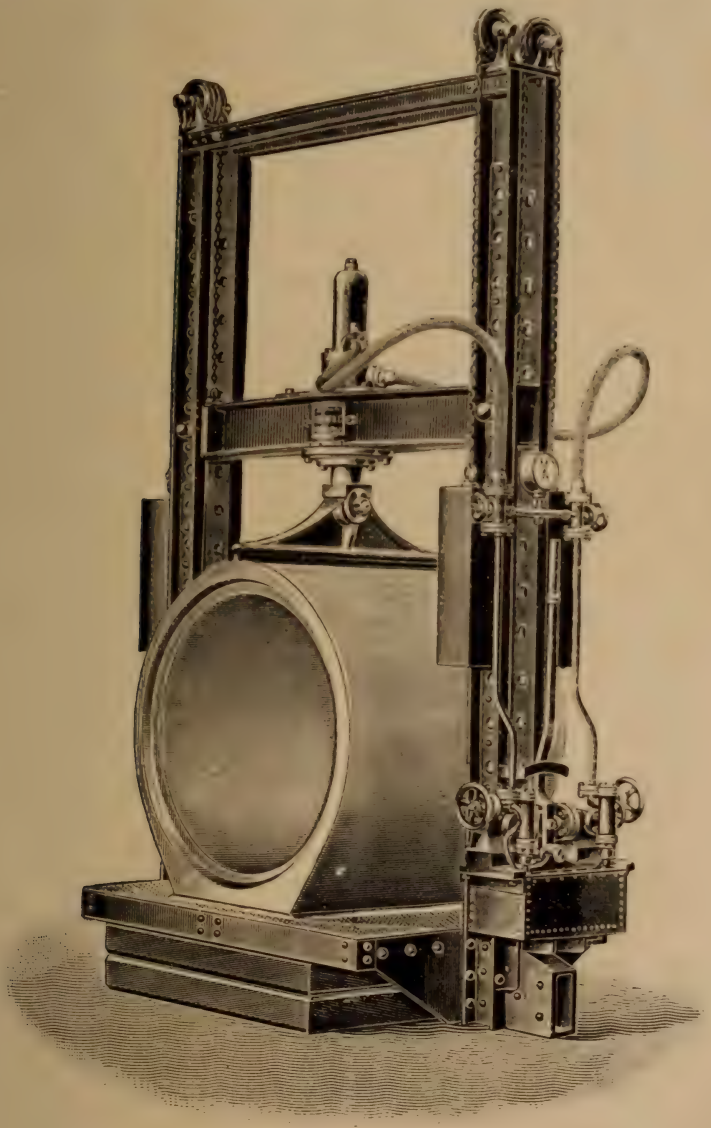

Fig. 7.

is a hydraulic press having a capacity of 10.000 kg. The piston is fixed in the upper cross-beam which is counter-balanced by weights, and adjusted by bolts, so as to allow of the testing of pipes 2 meters high. The vertical pressure is transferred to the pipe by means of an iron traverse directly connected to the piston and having an 18 cm stroke. The cylinder of the press communicates with the pumping device placed at the bottom of the frame and the stroke of the piston is governed by hand-regulated valves.

The pressure is indicated by a gauge attached to the pressure pipe and equipped with two independent gauge mechanisms fitted with scales. Another control may be had by attaching a second pressure gauge instead of the relief screw of the cylinder. The liquid used is glycerine.

b) The press of the Königliches Materialprüfungsamt in Groß-Lichterfelde, West (fig. 8). The press

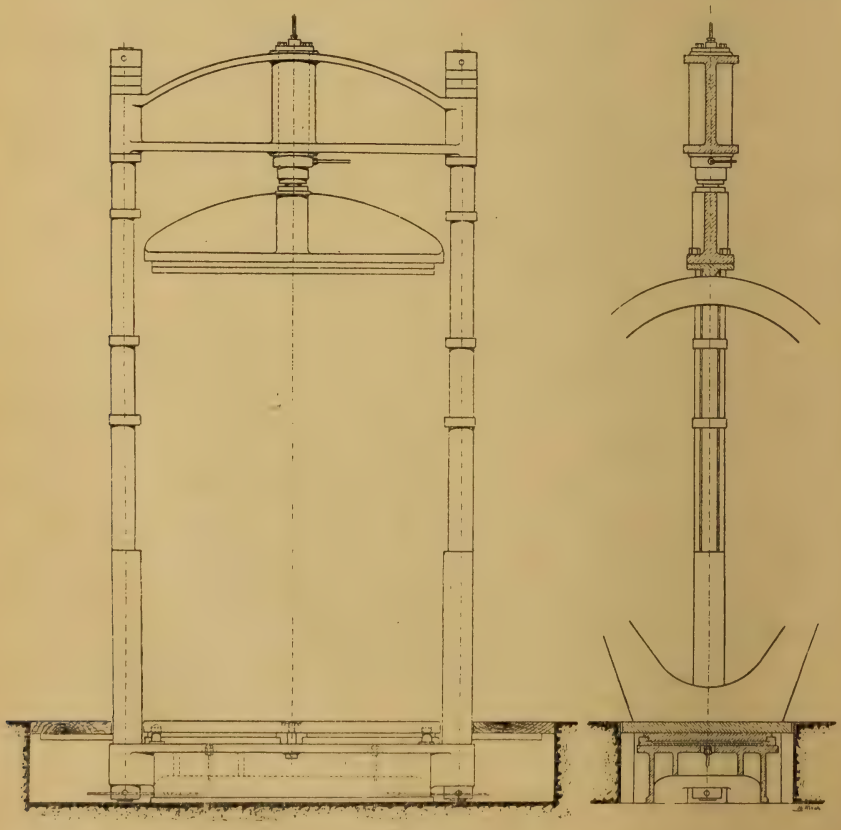

Fig. 8.

designed by Martens was built by A. Borsig, of Tegel; its greatest capacity is 20.000 kg. The pressure here is also hydraulic pressure obtained from the high-pressure hydraulic drains (with accumulators) of the Institution and transferred to the pipe centre by means of an iron cross-beam and thence through the pipe to the moveable foundation plate. The foundation plate rests on the covers of two large measuring boxes, the water pressure in the same being measured by gauges and recorded graphically by similar instruments, placed on the valve table close to the machine.

The valve-gear allows of a slight stroke of the piston and the pressure-head; as also of a quick or slow movement if desired.

The upper cross-beam of the press is moveable and is carried by slotted and interchangeable pipes of different lengths sliding on the tie rods.

(See also: „Denkschrift über das königliche Materialprüfungsamt in Groß-Lichterfelde“ pp. 350—351.)

c) The press of municipal-building-inspector Fried of Barmen. Koenen was made on the assumption that the extent of deformation provided a means of estimating the strength of pipes. This induced Fried to construct a press in combination with apparatus for measuring the extent to which pipes could be bent (fig. 9).

The press is a triple-arm lever-press with 1 to 100 gear. On the third lever is mounted the weight to be applied which is set in action by means of chain and chain wheels with skew-gear, the result being that no load need be applied by hand. As the pressure lever can be brought either into a horizontal or inclined position, the load moments can both be varied and a special scale introduced by means of which a pendulum indicator shows the amount of load applied. An adjusting mechanism enables the weight lever to be kept horizontal in all positions. With the first lever which is armed with the pressure block, is connected the indicating mechanism and the deformations are transferred from this to the diagram as ordinates; the abscissae give the values of the load.

It takes about a minute to make a diagram and the amount of force needed is slight.

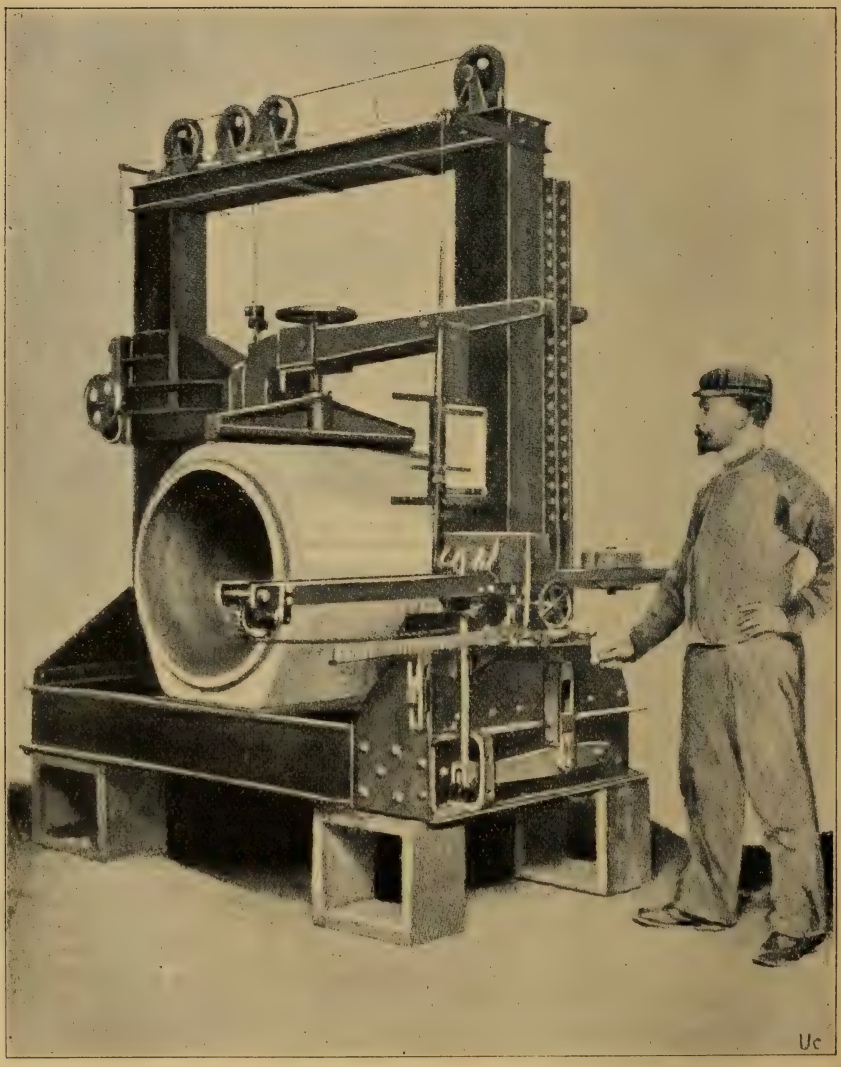

Fig. 9.

Fried is of the opinion that by means of his apparatus the degree of deformation with a given load supplies an adequate indication of the reliability of the pipe for practical requirements he,

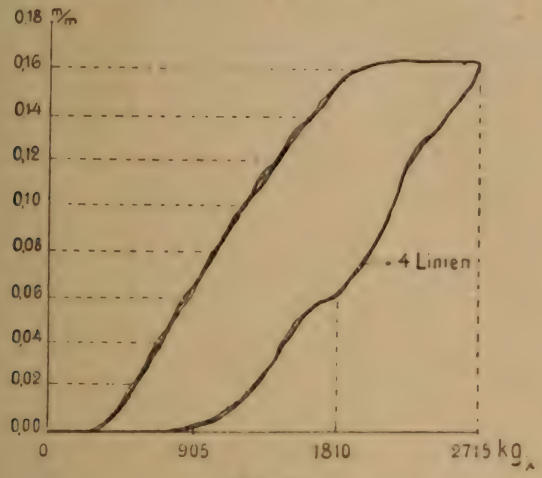

Fig. 10.

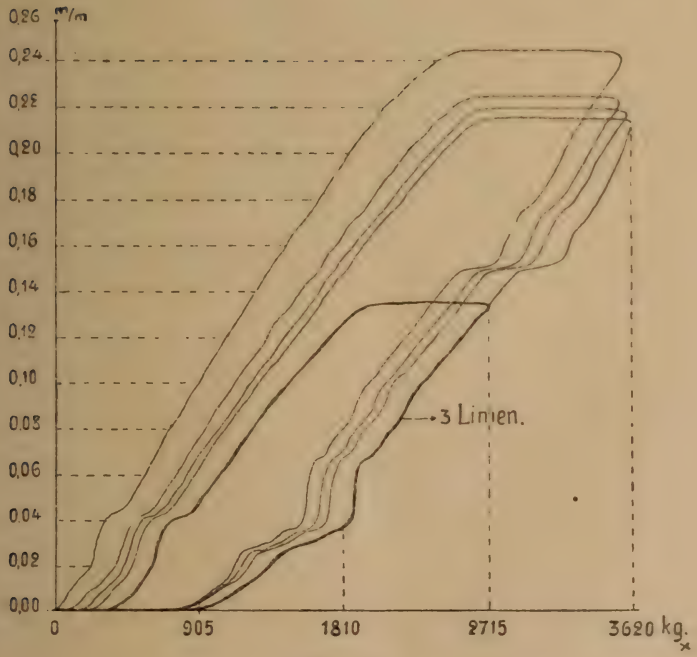

Fig. 11.

however, states that pipes from different factories should be compared with one another for the purpose of enabling comparative figures to be ascertained and in this case they should be loaded until fracture ensues.

The experience of several years has, however, shown that it is not possible to judge of the strength of pipes by the extent to which they will bend. Age, moisture, warmth, composition,

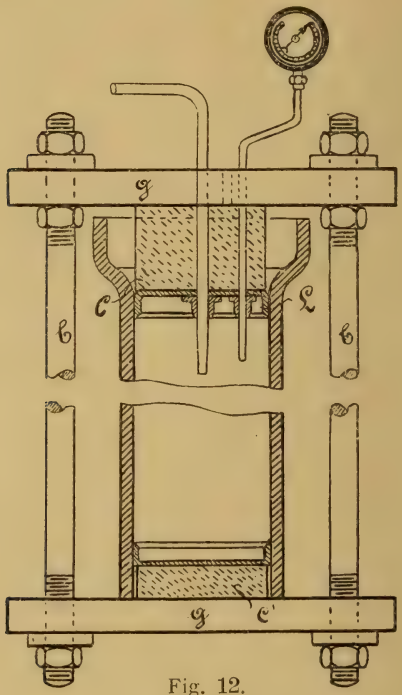

Fig. 12.

materials, percentage of water and other circumstances influence the degree of elasticity of cement pipes to such an extent that the degree to which they bend does not enable any satisfactory conclusion to be drawn regarding their strength.

Although, experiments of this kind possess a theoretic value only, they are worth mentioning.

Building-Inspector Fried made a communication to the general meeting of the German Beton Verein in 1905 regarding the results of some experiments he had made and gave a diagram reproduced in figures 10 and 11.

Fig. 10 illustrates a diagram obtained by repeated loading of a cylindrical tube of 80 cm. in diameter which shows that the pipe is sufficiently strong for the load.

Fig. 11 gives the diagram of a pipe of the same size which gave curves continnally varying from one another, which shows that the pipe did not possess the strength needed for the purposes to which it was to be applied.

d) Rudeloff's arrangement for testing for internal pressure is shown in fig. 12. Fig. 13 shows the arrangement for very large pipes. The yokes *g* which are maintained in position by the tie-rods *b* exercise no actual pressure on the pipe but merely serve as supports for the stoppers *C* which are packed against the pipe by means of rubber or leather expansion cups *L*.

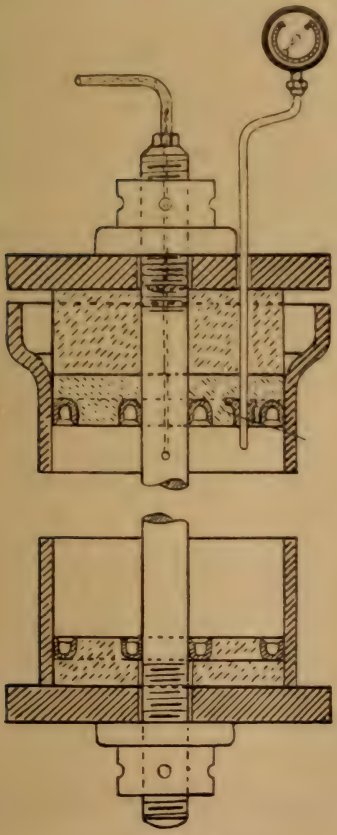

Fig. 13.

2. Recording instruments.

When testing the compressive strength of pipes measuring instruments of precision (mirror apparatus etc.) will as a rule not be used but simple instruments, easily mounted and dismounted, which do not cost much, and which can be constructed on the spot from material at hand.

Such simple apparatus has been designed by the Institution for testing materials in Groß-Lichterfelde. Two methods of construction are represented in fig. 14.

The apparatus consists of two thin wooden laths, between which is arranged a small roller with pointer attached, which is free to turn on its axis. The laths are pressed against the roller by

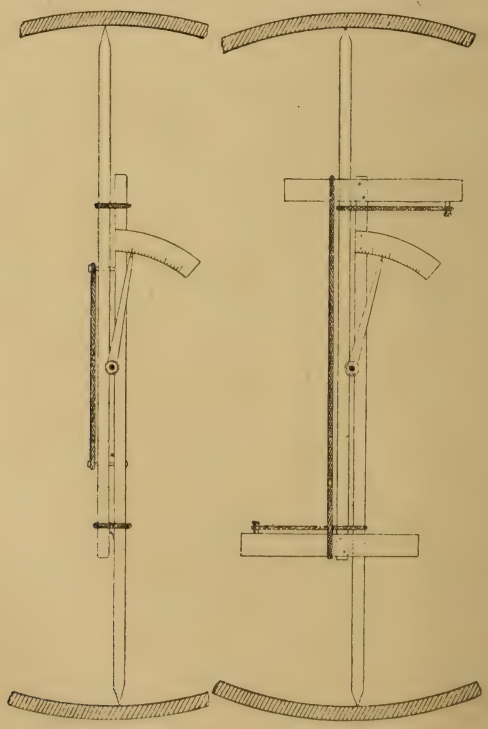

Fig. 14.

means of thin rubber tubes and made elastic longitudinally by stretched rubber tubes, which readily support the ends of the laths against the walls of the pipe.

When compressing the pipe the laths are displaced in opposite directions and the small roller turns, thus allowing the pointer to indicate by a considerable change of position a very small actual displacement (compression). The diameter of the roller easily admits the proportion of 1 to 250 or more between the actual and indicated displacement.

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 18.

Group C

ON THE METHODS OF TESTING THE PROTECTIVE POWER OF PAINTS USED ON METALLIC STRUCTURES.

BY **E. EBERT**, KÖNIGL. BAYER.
REGIERUNGSRAT IN MUNICH.
(TRANSLATED BY R. N. LUCAS.)

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On the methods of testing the protective power of paints used on metallic structures.

By **E. Ebert,** kgl. bayer. Regierungsrat.

Translated by R. N. Lucas.

There is no doubt whatever that the question of protecting iron employed for building purposes against rust and other prejudicial chemicial influences, is of very great industrial importance. An enormous amount of capital is sunk in all existing railways, the preservation and upkeep of which is very largely influenced by the effectiveness of means for preventing rusting.

Unfortunately even at the present day we are not yet in possession of any means which will enable us to form a rapid judgment as to the relative merits of different processes. It almost seems to me as if no such tests possessing practical value can be found.

Experiments which have hitherto been made for this purpose in laboratories prove almost absolute failures in practical employment, except in so far as they are concerned with the substances composing the compositions made use of, such for example as cinseed oil varnish.

Tests that have been made on a large scale enable conclusions to be reached as regards the manner in which the composition is employed and whether different compositions give similar results when equally exposed to the weather, but otherwise the

comparative value of the results is doubtful. That the desired end is difficult to attain is shewn by the fact that practical tests made by various people with the same rust-protecting composition, give rise to the most contradictory conclusions, a fact which is well known to persons entrusted with the preservation of iron buildings and structures. I also believe that the results attained in France confirm my view and I accordingly come to raise the question whether it is in the interest of manufacturers of anti-rusting preparations and those who use them to proceed in this manner? I believe that this question should be answered in the negative. And for the following reasons: Testing of building materials is in the first place conditioned by the demands made in regard to the durability and carrying capacity of the material. The formation of a decision on industrial grounds can only take place after the requirements have been fulfilled after the materials have remained protected for a certain length of time. If the effect of the protecting composition is not under observation or contro after they have been employed, it is to be feared that after a time the prejudicial effects on the durability of the structure will make themselves felt. The anti-rust compositions, however, are almost exclusively employed to protect iron buildings against atmospheric influences and their prejudicial effects. After substances have been employed they remain continuously accessible when the fundamental principle of the technology of iron is observed, namely, that all surfaces exposed to air and moisture must be so arranged as to be accessible to observation and are treated with the necessary anti-rust composition. If this is done, the comparative value of the various compositions which have been introduced with such promising names, can be rightly appraised in spite of their numerous testimonials without invoking any special assistance. If the observations are conducted unintelligently all tests of the quality of the materials employed are useless and their durability and general behaviour is dependent merely on chance, which may have the effect of conferring an apparent superiority upon what is really the inferior article.

The expert will invariably test any anti-rusting composition which he comes across with care and in the first instance on a small scale, preferably in juxtaposition with some well known composition which has proved its value. On the other hand the manu-

facturer should be informed that his material is not only being subjected to this test but is to be used on an extended scale; his activity should consequently be directed to surpass the existing anti-rust compositions in value and industrial applicability.

Where the possibility exists that an anti-rusting composition may be encountered or get into the market which only does not affect the rust but exercises a prejudicial effect upon the metal, or where it does the latter while doing the former, it of course becomes a duty just as when testing any other construction of material, to test its constituents previously. Hitherto however, I have not learned from personal experience nor from anything that had been published whether any of our well known anti-rust preparations produces such an effect.

The view previously entertained by many manufacturers that the formation of rust tended to promote further oxidation by chemical means and that therefore preparations containing oxide of iron exercised a prejudicial effect, has as the result of extended experiment, been fully and completely disproved.

Experiments with actual iron and rust have proved that if it is finely ground and rubbed up with linseed oil varnish it produces no prejudicial effect upon iron to which it is applied. If, however, rust has formed upon iron it acts very much like the sponge in virtue of its porosity, collects the water, retains it and so considerably promotes the further rusting of the metal.

This phenomenon has been long familiar to experienced constructors and is the reason why they always take care to build in such a way and to select the colour of the paint employed so that any formation of rust can be easily recognised the instant that it takes place. Pigments and binding materials are separate substances and do not form chemical combinations with one another. The pigment is exactly the same after the paint has dried as it was before being mixed with the liquid oil. The dried oil, however, closes the pigment as if in a sheath and does not itself attack the iron.*) For example a coating of paint consisting of

*) (Is not this an error? The view generally prevailing in this country is that the oils take from the oxides with which they are mixed to form paints some of the oxygen required for their solidification. R. N. L.)

linseed oil and white lead can be placed for weeks in dilute nitric acid without the least trace of metal going into solution, though as is well known pure white lead is rapidly dissolved by dilute nitric acid. We see therefore that the quality of an oil paint depends in the first place upon the quality of the linseed oil employed in its composition. The pigment itself plays a quite subordinate role as long as it is surrounded by oil. If however, it happens to be laid bare by mechanical or physical influences it may have the effect of rapidly destroying the paint. Neutral acid-proof pigments, therefore, are probably most suitable for use in such circumstances where there are no objections to the class of colour.

Further, according to the above mentioned experiments, there can be no objection, particularly where the colour does not matter, as in the case of body coats, to leave the pigments out altogether.

The information which has been supplied to us from France, Germany, Russia and other countries, do not lead to the conclusion that there is any general agreement as to any particular anti-rust composition being superior to the other. On the contrary, the views differ very considerably. Agreement, however, prevails that the testing of such compositions prior to their use would not lead to the results desired by the Institute.

None of the anti-rust preparations with so-called acid-proof components have proved, according to my experience, adequately to resist the action of the acids of sulphur which are contained in the smoke of locomotives.

If we are in a position to procure the materials from our own manufacture, namely the linseed oil and pigments and to mix them properly, there can be no doubt that reliable methods can be adopted to ascertain whether the materials are of good quality or not. The process, however, is only applicable in a small number of instances and considering the extensive character of the manufactures connected with the production of anti-rusting compositions, is hardly to be recommended. If, however, we desire to produce the anti-rust material on a large scale it may be advisable to procure the materials as is done in building contracts and to ensure that they possess the necessary good qualities. The durability of the layer of preparation in contact with the metal depends upon the quality of the first coating of body-colour; it

is in addition largely determined by observing the principle that it should only be applied in warm, dry weather, and only to surfaces which are themselves dry. Unfortunately the preparation of the body coat is not always carried out with a degree of attention that should be bestowed upon it, but is often done by incapable workmen as unskilled labour, particularly when the end of the contract period is approaching. In addition, the cleaning of the surface of the iron is often not properly done, with the result that the body coats do not produce the proper effect. In a short time they fall off altogether.

Though the importance of cleaning has been pointed out it does not, however, follow that the iron surfaces must be absolutely bright Experiments which were carried out in the year from 1892 to 1896 in order to ascertain what was the effect of cleaning, have proved that it is fully sufficient in order to free the iron from all impurities to go over it with a wire brush or scraper, or if need be to employ a hammer to knock off any adhering scale. The layer of oxide or rust which remains after this treatment does no harm and is in fact serviceable, as the preparation adheres better to it than it would to a bright and polished iron surface. The management of the (name illegible) railways for many years maintained that the iron before being treated with the compositions should be cleaned bright in dilute hydrochloric acid, then inserted into lime water, washed in hot water, warmed up to a high temperature and after the very rapid drying which then results painted over with linseed oil varnish. Nothing can be alleged against this process as a matter of principle when carried out on a large scale by competent workmen. It is, however, a very different matter when on small work and with inadequate plant the same process is attempted usually unwillingly. In such cases there is always the danger that the hydrochloric acid which has penetrated into the interstices of the iron will remain there and produce a deleterious effect. Further the experiments above referred have shewn that the methods adopted in the Bavarian State Railways of using a body coat of linseed oil varnish without pigment has not caused any trouble. The articles are also subjected to examination after receiving the first coating when lying about waiting to be removed by the consignees. Von Tetmeyer and Dr. Landolt have recommended this process on various occasions. Objections have been

raised by various parties that they have not been adequately supported. Taking into consideration the observations of the members of the international commission, I am prepared to answer question 18 in agreement with them to the effect that the durability of the anti-rust preparation depends upon the quality of the linseed oil employed, and we have accordingly devoted the greatest attention to the preparation of the latter. For deciding the quality of the linseed oil, it is very desirable that we should be in possession of some process which will enable us to ascertain quickly, simply and reliably what its quality is, even when already mixed with pigments.

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REPORT ON PROBLEM 18.

Group C

ON THE METHODS OF TESTING THE PROTECTIVE POWER OF PAINTS USED ON METALLIC STRUCTURES.

RAW AND BOILED LIN-SEED OIL

BY **A. GRITTNER**, CHIEF ANALYTICAL CHEMIST TO THE HUNGARIAN STATE RAILWAYS, BUDAPESTH.
(TRANSLATED BY CH. CAPITO, LONDON.)

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Raw and Boiled Linseed Oil.

Report by **A. Grittner**, Chief Analytical Chemist to the Hungarian State Railways.

(Translated by Ch. Capito, London.)

Raw Linseed Oil.

Linseed oil is produced by cold or hot expression of the seeds of the common flax or lint, Linum usitatissimum. The cold pressed oil, „cold-drawn oil", is of a yellow colour, while the hot pressed oil is of a yellow to brownish-yellow colour, according to the temperature to which the ground seeds have been heated. The cold drawn oil is exclusively used for food, whereas the hot pressed oil is extensively applied to technical purposes.

The oil when leaving the presses runs into large reservoirs, where it settles and remains until it is clear. The complete settling in large depositing-tanks takes a long time, and it is therefore more expedient when, after some time, a certain depth has become clear to let the oil run out and filter it through filter-presses and then put it on the market. The oil requires only 6—8 weeks to settle, and not 1—2 years as is sometimes stated to be necessary when it is to be used for the manufacture of boiled oil. The oil from the filter-presses will after a time still leave some deposit, which however is not of great importance for boiling. To let the oil remain for 1 to 2 years in the settling tanks would require for even moderate sized works a large space and therefore also a large capital outlay. If it is to be used for varnish, the linseed oil must either be left standing for 1 or 2 years or the mucilaginous parts must be got rid of by heating.

For the purpose of examining the purity of linseed oil, the following tests should be made:

1. Specific Gravity. The specific density should always be determined by means of the Pyknometer and at 15° C. It is very important that this temperature should be agreed upon, as the specific gravity is very often determined at other temperatures. The specific gravity at any temperature can be reduced to that at 15° C. by considering the expansion of the oil; the required correction is according to Holde[1]) 0·00068 per degree, and according to Allen 0·00064; the corrected values however do not perfectly agree with those found by direct measurement. I do not consider that the hydrometer method is sufficiently accurate and therefore it should only be used for factory purposes.

The specific gravity of linseed oil is differently given by different authorities. A. H. Gill and A. C. Lamb[2]) examined pure American linseed oil, and found the specific gravity at + 15° C. to be between 0·931 and 0·938; G. de Negri and G. Fabris[3]) give it between 0·9290 and 0·9352; Filsinger[4]) found that the specific gravity of linseed oil from various sources varies between 0·9305 and 0·9370 at 17·5° C.

I have examined linseed oils from various manufacturers, made for technical purposes, and also cold-pressed oil prepared by myself from Hungarian linseeds. I found the specific gravity of these oils to be between 0·9284 and 0·9326 at + 15° C.

Although linseed oil is denser than the oils which are generally used for its adulteration, no conclusion can be drawn from the specific gravity, unless it is found to be below 0·928 or above 0·938. A lower density suggests an addition of rape-seed oil, mustard oil, cotton-seed oil or mineral oil; higher density would be caused by rosin, rosin oil, or over-heated linseed oil or by addinga siccative.

2. Iodine Constant. The iodine constant of linseed oil is one of the most important, but after all perhaps also the least settled constant. The great variety of values given for this constant

[1]) Untersuchung der Schmiermittel. Berlin 1897.
[2]) Chemisches Zentralblatt 1899. I, 644.
[3]) Zeitschrift für analytische Chemie, 33, 547.
[4]) Chemiker-Zeitung, 18, 1005.

is chiefly due to the different ways in which the operation of determining it has been carried out. It must however be admitted that the origin of the oil exercises a great influence on the value of this constant.

The iodine constant indicates the percentage of jodine which will combine with a linseed oil. As is well known, iodine will only combine with the unsaturated oils, and can therefore be considered as a measure of the amount of unsaturated fatty acids present in the oil.

It was Hübl[1]) who worked out the method for determining the iodine constant. According to his prescription, from 0·1 to 0·2 grm. of drying oil should be weighed and poured into a bottle with a well ground stopper, and then dissolved in 15 cm^3 of chloroform; 25 cm^3 of iodine solution is then added. If the liquid is decolorised within a short time, another 25 cm^3 of iodine solution is to be added, and the colour of the liquid must remain strongly brown after the lapse of two hours. The reaction is then finished. Twenty cm^3 of a ten per cent solution of potassium iodide, and 150 cm^3 of water are next added, and finally the excess of iodine is titrated.

This method has been the object of many tests, and it appears that the method suggested by Hübl contains many errors. It has been proved that Hübl's iodine solution is not stable, and also that the experimental conditions, under which the method has to be executed, must be exactly fixed. Gantter[2]) believes that the reason why Hübl has obtained so many different results is due to the mercuric chloride which is added and which, notwithstanding the large excess of iodine, affects the result according to the quantity used. Although the results obtained by Gantter confirm this statement, the cause of the inconsistent results can nevertheless not be due to that, as the amount of mercuric chloride which is used is fixed. Gantter's proposal, not to use mercuric chloride and to dissolve the iodine as well as the fat in carbon tetrachloride seems not to be applicable; this method is also an arbitrary one, and the results do not agree with others.

D. Holde[3]) has thoroughly examined the Hübl method, and has proved that the results depend on the age as well as on the

[1]) Dingler. 253, 281.

[2]) Zeitschrift f. anal. Chemie 1893, 178.

[3]) Mitteilungen d. k. Versuchsstationen Berlin 1891, 81.

excess of iodine solution, and that the duration of the action must also be considered. Holde takes 0·2 grm. of drying oil and requires for a two hours' action an excess of from 65 to 75 per cent iodine, before the back-titration takes place. Holde does not always obtain good concordant results. He obtained for the same linseed oil the iodine constants 172·1 and 181·8, although in the first case there was 72 per cent excess of iodine, and in the second case 78 per cent excess. The same iodine solution gave on the same day with a 78 per cent excess, a constant of 181·8, and with a 68 per cent excess, only 175·5. The iodine solution must according to Holde not be more than 8 days old. It appears that a two hours' action is not sufficient, notwithstanding the great excess of iodine solution. In the Proceedings of the Royal Technical Research Laboratories of Berlin 1896, 319 will be found some remarks to the effect that if concordant results are to be obtained, a 24 hours' action is necessary. D. Holde[1]) has later laid down the following rule for finding the iodine constant: 0·2 grm of oil is to be taken together with 25 ccm of iodine solution and the iodine constant is then to be determined after a 24 hours' action. If it be necessary to make the test in a shorter time, an excess of 75 per cent of iodine must be used, and the time of action may then be reduced to two hours.

According to his own investigations, H. Bremer[2]) finds that the Hübl method is a very exact one. For drying oils, he finds that an 18 hours' action and only an excess of 30 per cent of iodine are required. The temperature is also of importance, for when everything else remains the same, the results vary if the temperature is below 15° C. or above 30° C.

A. Katz[3]) caused iodine solution to act on raw and boiled linseed oil for 2, 6, 12, 24, and 48 hours in the absence of light. He found the iodine value to be already constant after a lapse of 24 hours, and recommended that the action should be allowed to proceed in the dark. He quotes 182 as the mean iodine value for raw linseed oil, and 173 for that of boiled oil.

[1]) Chem. Revue für Harz- und Fettindustrie, 5, 41.

[2]) Forschungsberichte über Lebensmitteluntersuchung, 1, 318.

[3]) Forschungsberichte über Lebensmitteluntersuchung, 2, 203.

Rowland Williams[1]) has used an excess of 200 per cent of iodine and allowed the Hübl iodine solution to act for 18—20 hours. Several hundred samples of linseed oil have been tested by this method and, when not adulterated, gave no iodine constant below 180; in most cases the constants were near 190 and even above. From 53 Baltic samples, he found a mean iodine constant of 188·5.

The following standard rule for finding the iodine constant has been suggested by R. Henriques[2]) viz: The time of reaction should be 24 hours and the test sample should be protected from light. There should be at least an excess of 60 per cent of iodine. The strength of the iodine solution, originally N/5, must not sink below N/8. The strength of the iodine solution should be ascertained by a blank test, two parallel determinations being always carried out side by side.

B. A. Van Ketel and A. C. Antusch[3]) consider that the iodine value affords very valuable means for determining the presence of foreign oils in a linseed oil, as it can be regarded as approximately constant. They take, like Rowland Williams, the iodine constant between 180 and 190, and regard a linseed oil, which has a lower constant, as adulterated This statement must be considered as hazardous, especially as the authors used extracted oils in their trials. Their conclusions cannot be applied to expressed oils without caution, as H. Mastbaum[4]) has shown that a separation of the mixed glycerides, which are naturally contained in all oils takes place during the pressure. The expressed lighter liquid glycerides, generally belong to the unsaturated acids, and show a higher iodine constant than those remaining in the pressed cakes. The origin of the linseed oil also plays an important part in this question.

F. Ulzer[5]) recommends also the normal methods and suggests that the iodine constant should be determined with an excess of 100 per cent and a 6 hours' action. The iodine solution can

[1]) Chemisches Zentralblatt 1896, I, 225.
[2]) Chem. Rev. f. Harz- und Fett-Ind., 4, 80.
[3]) Zeitschrift f. ang. Chemie 1896, 581.
[4]) Zeitschrift f. ang. Chemie 1896, 719.
[5]) Chem. Zentralblatt 1898, I, 150.

be used as long as 25 ccm of it are capable of decomposing 35 ccm of $^1/_{10}$ normal thiosulphate solution.

A. H. Gill and A. C. Lamb[1]) have examined American linseed oils and have found after 4 hours' action, iodine constants varying from 160 to 178·8; after 18 hours, the constants varied from 160 to 180. It thus appears that in this case the iodine constants remain the same notwithstanding the different durations of action; this fact can only be attributed to the use of a larger excess of iodine.

It will be gathered from this somewhat incomplete collection of data that very different methods are used for determining the iodine constant; and it is certain that it is at least partly to this circumstance that we may ascribe the unsatisfactory lack of agreement in the values found. We may therefore say that we do not know at present how high the iodine constant should be of unadulterated linseed oils from different sources. There is no doubt that the source influences the iodine constant, as J. J. A. Wijs[2]) has shown. Besides the latter author, G. de Negri and G. Fabris[3]) give the iodine constant as 157—187; M. Tortelli and R. Ruggeri[4]) give the constants 173·4—179·4 for Italian linseed oils; H. F. Vulté and Lily Logan[5]) in publishing the iodine constants of various oils, give the constants for linseed oil as 155·1—155·5; B. Sjollema[6]) found the constants 165—185 for linseed oil prepared by himself; J. A. Roelofsen[7]) determined the iodine constant for various oils and found a constant 154·1 for linseed oil.

The different results which have been obtained by different methods of working and with iodine solutions of different ages, have caused various modifications in the original process to be suggested; the object being either to improve the Hübl reagent, or to devise some totally different iodine solution with the aid of which more concordant results should be obtained.

[1]) Chem. Zentralblatt 1899, I, 644.
[2]) Chem. Revue d. Harz- und Fett-Ind., 6, 29.
[3]) Zeitschrift f. anal. Chem., 33, 547.
[4]) Chem. Zentralblatt 1900, II, 598.
[5]) Chem. Zentralblatt 1901, I, 1179.
[6]) Zeit. f. Nahr.- u. Genußm. 6, 631.
[7]) Chem. Zentralblatt 1894, II, 255.

Merkling[1]) has suggested keeping the iodine and the mercuric chloride solution separate, and not mixing them until required for use. Fahrion[2]) has made the same suggestions as Merkling, but at the same time proposed to use methyl instead of ethyl alcohol.

H. Schweitzer and E. Lungwitz[3]) found by their researches, that when using the Hübl method, there is besides the addition of iodine also a substitution of iodine, which takes place simultaneously with the addition and increases with the duration of the reaction. One must endeavour to find a solution, with which the addition will be more complete and no substitution will take place. According to their view, this can be done by using carbon bisulphide or tetrachloride instead of alcohol, as these substances are not attacked by the iodine, and the strength of the iodine solution will therefore remain unaltered. If carbon bisulphide be used, then no substitution takes place, as the authors found when testing lard, cottonseed oil, and oleic acid; in the case of oleic acid, the iodine constants were even higher than the theoretical one, for which no explanation can be given.

Instead of chloroform, K. Farnsteiner[4]) uses benzene free from thiophen, which does not influence the iodine solution.

Waller[5]) considers that Hübl's iodine solution is stable when using an alcohol which is as free as possible from water, and is saturated with hydrochloric acid gas; or one may add concentrated hydrochloric acid to the iodine solution.

R. Pelgry[6]) recommends the use of Waller's solution. Analysis of 13 different oils with both Waller's and Hübl's solutions gave results which agreed within the limits of error. Greater differences were only shown in the case of rosin oil and an oil containing train oil, which by Waller's method gave 4·8 and 1·6 less than by Hübl's method. The time of action was 24 hours.

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[1]) Chem.-Ztg. 1887, 22.
[2]) Chem.-Ztg. 1891, 1791; 1892, 862.
[3]) Chem. Zentralblatt 1895, I, 812; 1896, I. 515.
[4]) Zeitschrift f. Nahrungs- und Genußmittel 1898, 729.
[5]) Chemiker-Zeitung 1895, 1786.
[6]) Chem. Revue für Harz- und Fettindustrie 4, 78.

The use of alcoholic iodine monochloride solution for determining the iodine constant was first proposed by J. Ephraim[1]). The results obtained with this solution agree well with those obtained with the Hübl-solution. This solution shows however a somewhat smaller stability than Hübl's; there is therefore no reason why it should be used, especially as the commercial iodine monochloride is rarely pure.

R. Seeliger[2]) uses a chloride of iodine solution which is made by dissolving 10 gms. of iodine in 100 gms. of tetrachloride of carbon, adding chlorine and filling up with alcohol to 250 cm^3 The results agree very well with Hübl.

J. J. Wijs[3]) uses a chloride of iodine solution in glacial acetic acid, which he prepares himself by leading chlorine into a solution of iodine in glacial acetic acid, or by dissolving trichloride of iodine in glacial acetic acid and adding 2 atoms of iodine.

C. Aschman[4]) uses an aqueous iodine chloride solution. Although his results do not differ widely from those of Hübl, such a liquid should not be employed, as oil dissolved in chloroform does not mix with it.

Although these proposed modifications of the Hübl-solution cannot be adopted, Wijs undoubtedly deserves the merit, by his valuable work, of having explained the mechanism of the Hübl process.

Finally, it should be mentioned that B. Sjollema[5]) by using the Wijs-solution came to the conclusion that the iodine constant depends on the purity of the chemicals. Differences of 11 units were observed with the same linseed oil treated with chloride of iodine solutions of different origin although the titre was the same. Even solutions from the same iodine and the same acetic acid, but in one case prepared with gaseous chlorine, and in another case with trichloride of iodine, gave a difference of two units.

As has been mentioned, a standard method for ascertaining the iodine constant does not exist. Most authorities agree that a large

[1]) Zeitschrift f. angew. Chemie 1895, 254.
[2]) Pharmazeut. Zentralblatt 35, 89.
[3]) Zeitschrift f. angew. Chemie 1898, 291.
[4]) Chemiker-Zeitung 1898, 59.
[5]) Zeitschrift f. Nahrungs- und Genußmittel 6, 631.

excess of iodine and a long duration of action are the chief conditions required for obtaining a correct iodine constant.

In my own analyses, I have kept to the preparation of the solution as prescribed by Hübl. The iodine and mercury solutions were freshly made, and used after standing for 24 hours. I weighed 0·2—0·3 gm. of linseed oil and dissolved it in 15 ccm of chloroform, added 50 ccm of iodine solution and left the mixture protected from light for 24 hours. The excess of iodine was at least 75 per cent. A blank test was also carried out simultaneously, consisting of 15 ccm of chloroform and 50 ccm of iodine solution; this was done for the purpose of being able to take into consideration the error which arises when the iodine solution acts on the chloroform. After a lapse of 24 hours, 50 ccm of 10% iodide of potassium solution were added; this large amount of iodide of potassium was used in order to prevent any separation of iodide of mercury. 150 ccm of water were then added and the excess of iodine was titrated back. Some starch-paste was added towards the end of the titration. In this manner I have tried 16 linseed oils obtained from factories, where they were to be converted into boiled oils. The iodine constants of these samples were found to vary from 159 to 173. Four linseed oils from Hungarian linseeds, which I expressed myself, gave iodine constants from 174 to 178. I must therefore contradict Rowland Williams, who maintains that no linseed oil, which is not adulterated, can have an iodine constant below 180; the linseeds which I pressed were completely free from foreign seeds. If the observations of H. Mastbaum are to be taken as correct, then hot pressed oils must give a still smaller constant. It must partly be ascribed to the latter circumstance when commercial linseed oils give still lower constants. It is certainly also necessary to take into consideration the fact that the linseeds which are used for manufacturing purposes, always include a percentage of foreign seeds, which however is so small that no adulteration can be shown by analysis. The question about the iodine constant can therefore only be solved by examining seeds from various sources such as are employed in the manufacture of oil; the seeds being carefully freed from foreign seed, and then expressed in the laboratory. This task is the more important, as by uniform work, the causes of the present differences in the iodine constants may finally be cleared up.

3. Acid Constant. The acid constant is the number of milligrams of hydrate of potash per gram of oil, which is required for neutralising the free fatty acids contained in the oil.

The acid constant is found by taking 5—10 gms. of oil and dissolving it in ether-alcohol 1 : 1, and titrating with an alcoholic solution of caustic potash (about N/10), adding phenolphthalein as indicator. A watery solution of potash may also be used, which has the advantage, that it does not require to be continuously controlled as the alcoholic solution of potash requires. The alcoholic solution possesses however the advantage, that by using it, the ether-alcohol will not be diluted with water, and by the distillation of the residuum a quantity of aqueous alcohol will not accumulate.

The acid constants of commercial linseed oils which I have examined vary from 1·1 to 3·1, while oils freshly expressed by myself only gave 0·2—0·7. The acid constant of linseed oil is thus very small and this has also been found by Nördlinger[1]) and Lewkowitsch[2]). A high acid constant indicates the presence of rosin.

4. Saponification Constant. The saponification constant, also called the Köttstorfer constant, is the number of milligrams of hydrate of potash required to saponify 1 gram of oil completely.

The determination of the saponification constant requires an accurately prepared N/2 hydrochloric acid and an alcoholic solution of caustic potash of about N/2 strength. 1·5 to 2·0 gms. of oil are treated with 25 ccm alcoholic potash solution and saponified. The saponification takes place on a boiling water bath. When the saponification is completed, the surplus of potash solution is titrated back, after adding phenolphthalein, with N/2 hydrochloric acid. As the strength of the potash solution changes, it must be checked before each analysis, and the same conditions which are observed when determining the saponification constant, must be strictly adhered to. Only glass-bottles made of resistant glass should be used, the best is Jena-glass; there are some kinds of glass, which are attacked by the caustic potash, and which will therefore influence the accuracy of the result.

[1]) Zeitschrift f. anal. Chemie 1889, 183.

[2]) Chem. Revue f. Harz- und Fettindustrie 1898, 211.

R. Henriques[1]) has shown that the saponification constant can be determined by a cold method. According to his specification, 3 to 4 gms. of oil should be weighed and dissolved in 25 ccm of petroleum ether and then 25 ccm normal alcoholic potash solution added. The test-sample is shaken and then left standing for 12 hours; after the lapse of this time, it is titrated back with normal hydrochloric acid. When using this method, the alcoholic potash solution must be prepared by letting the caustic potash be boiled with 95 per cent alcohol under a reflux condenser. When dissolved, the solution is left standing for 24 hours and then filtered. This precaution must not be neglected, or else the petroleum ether will not mix with the alcoholic potash solution. If a solution be used in which the caustic potash was dissolved in water and then filled up with alcohol, the petroleum ether will separate from the solution; this however can be avoided, by adding 1—2 ccm of ether. This method possesses great advantages over the warm saponification; the alkaline solution after saponification will never be so dark as to make the back titration difficult. Any kind of glass may be used, as the alkaline solution hardly attacks the glass.

The saponification constant of linseed oil is rather high and ought to be at least 187. This figure agrees with my own results. I have found that the saponification constants of commercial linseed oils are from 187 to 194 and of oils expressed by myself from 190—192.

Linseed oil which is adulterated with rosin, rosin oil or mineral oil has a smaller saponification constant. Amsel[2]) considers that the presence of these oils can be shown for certain by the lower saponification constant and especially by the turbidity which is produced when water is added to the alcoholic soap solution; this he calls the „water reaction". The reason is that these substances are not or are only partly saponified and the watery soap solution will therefore become turbid. He goes still further, and concludes from an analysis which is recorded in table II under No. 4, that a linseed oil whose saponification constant is 183, which becomes milky by adding water, and whose iodine constant is only 112, must contain rape oil. If this linseed oil had been

[1]) Zeitschrift f. angew. Chemie 1895, 721.

[2]) Zeitschrift für angew. Chemie 1895, 733, 862.

adulterated with rape oil only, it could under no circumstances become milky by adding water.

W. Lippert[1]) has proved that the presence of rosin cannot always be shown by the water reaction, as various kinds of colophony exist which give a clear solution when water is added after the saponification. Even the presence of mineral oils cannot always be shown by the water reaction; for if the adulterant is a petroleum oil distilling between 160° and 175° C, the solution will not be milky unless 30 per cent of mineral oil is present at least, and the same is the case with rosin oil. Lippert quotes an analysis in which the solution only became milky on the addition of 20 per cent of rosin oil. One cannot therefore rely on the water reaction, so strongly recommended by Amsel, as the lower saponification constant after all only indicates that the linseed oil is adulterated. If the saponification constant is lower than 170, then only rosin oil and mineral oil can be present; if the acid constant be also high, then rosin is present, even if the saponification constant is higher than 170, but smaller than that found for linseed oil. The quantitative analysis of the unsaponified portion will disclose the nature of the adulteration.

5. Refraction. A. Müller[2]) introduced the refractometer into the analysis of fats and oils, and recommended it for the examination of butter. Jean[3]) attributed great importance to the refractometric examination of oils. The refractometer can according to R. Hefelmann and P. Mann[4]) also be used for judging linseed oils One might have thought that there would have been a simple relation between the iodine constant and the refractometer constant, and that oils with a high iodine constant would also have shown higher refraction. M. Weger[5]) has shown that the iodine constant and refractometer constant are not always in direct proportion to each other. When adulteration is out of the question, the refractometer constant can only indicate the manner in which the linseed oil has been treated, as a high refraction indicates heated or old oil. Further conclusions cannot be drawn from the constant.

[1]) Zeitschrift für angew. Chemie 1897, 306, 655.
[2]) Archiv der Pharmazie 1886, 210.
[3]) Comptes rendus 1889, 109.
[4]) Pharm. Zentralblatt 1895, 16, 685.
[5]) Zeitschrift für angew. Chemie 1899, 297, 330.

As rosin and rosin-oil have very high refractometer constants, it follows that their presence can be shown by means of the refractometer; thus a linseed oil adulterated with 20 per cent of rosin or rosin-oil gives, at 25° C., a refractometer constant of over 100.

The refractometer constants of linseed oils examined by me were determined, like Weger, at 40° C., and I have found for commercial oils constants from 72 to 73·5, and for oils expressed by myself I found the figures 73·5—73·8.

6. Absorption of Oxygen. It is a well known fact, that all fatty oils when exposed to the air become thick (resinified), due to absorption of oxygen. The resinification depends firstly on the nature of the oil, and certainly the oils which contain unsaturated fatty acids absorb oxygen quicker. As linseed oil contains linoleic acid, the absorption of oxygen is very rapid. The resinification also depends on the surface, the temperature and the light. The absorption of oxygen does not only consist in an addition of oxygen, but also in a deeper decomposition; the former action is however greater than the latter, while the increase in weight is much greater than the loss in weight. The latter is caused by the formation of volatile products, such as water, carbonic acid and lower fatty acids.

Clöez[1]) determined the absorption of oxygen of oils by exposing them in thin layers to the air at ordinary as well as at higher temperatures. According to his investigations, linseed oil increased 7 per cent in weight during the first 18 months and then lost in weight. R. Kissling[2]) repeated Clöez's experiments. He not only used the same weight but also the same surfaces of exposure, and found that at the temperature of the room, the increase in weight of linseed oil was 1·13 per cent after 10 days' exposure. Kissling's results prove that the figures for the absorption of oxygen are not free from objection, as the layer of oil is too thick and the innermost part is partly protected from oxygen; this is especially the case when the surface is covered with a skin which prevents the oxidation. Mulder[3]) painted the oil on plates

[1]) Bulletin de la société chimique de Paris 1865, 41.

[2]) Zeitschrift für angew. Chemie 1891, 395.

[3]) Chemie der austrocknenden Öle 1867.

so as to eliminate the objection to Clöez's method, but the layer of oil was still too thick; he found for linseed oil an increase in weight of only 11·9 per cent. This number is too low, because the maximum absorption was not reached on account of the thickness of the layer of oil.

Livache[1]) determined the absorption of oxygen by means of lead-powder (reduced from lead-salts by zinc) which he spread on a watch-glass and soaked in oil. In this manner the surface of the oil is supposed to be increased and the lead accelerates the absorption. Drying oils reached already after 3 days their maximum weight. Linseed oil showed an increase in weight of 14·3 per cent after the lapse of 2 days.

W. Bishop[2]) has modified Livache's method. He dissolved 0·02 gm. of manganese resinate in 1 gm. of oil and stirred it together with precipitated and ignited silica. He found an increase of 16·4 to 17·6 per cent by weight for French linseed oils, and for La Plata linseed oils, an increase of 15·0 to 15·45 per cent.

W. Lippert[3]) and M. Weger[4]) have made very exhaustive trials on the increase in weight of linseed oils by using very thin layers. Lippert employed tin-plates 18×10 cm and weighing 56 gms. According to his investigations, the increase in weight amounted to between 12 and 16 per cent. As the tin-plates were rather heavy he afterwards used glass-plates of 0·2 mm. thickness and 75 qcm surface, which only weighed 2·5 to 3·5 gms. On these plates he spread from 0·03 to 0·05 gm. of oil, i. e. 0·0004 to 0·0008 gm. per qcm. The oil was put on thinly and 1 cm from the edge, the plate being tilted at an angle of 30°, so that the surplus of oil could run down and be wiped off. The plate was then placed on the balance with the 1 cm clean edge downwards. Weger found that the average absorption of oxygen for different linseed oils, including such as had been treated, air-blown and heated, to be 15 per cent, and for pure linseed oils from different sources, he found constants from 15·1 to 19·9.

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[1]) Comptes rendus 1883, 96.
[2]) Chem. Zentralblatt 1896, I. 527.
[3]) Zeitschrift für angew. Chemie, 1898, 490, 507; 1899, 297, 330.
[4]) Zeitschrift für angew. Chemie, 1898, 412, 431; 1899, 540, 511; 1900, 133.

If the absorption of oxygen is high, then we may conclude that the linseed oil is fresh and has not been much exposed to the air. If however the oxidation constant be small, then the linseed oil is old and has been long exposed to the air, or, as shown by Lippert, it may be adulterated with an oil which only absorbs a small quantity of oxygen, as for instance mineral oil.

The rapidity with which the absorption of oxygen takes place is, like the time of drying, dependent on light, temperature and the dampness of the air. Weger found that the drying of linseed oil takes 3 to 8 days, and Amsel's[1]) statement that unadulterated linseed oil should dry in 3 days, cannot therefore be accepted. Amsel does not mention any results concerning the drying-time of linseed oil; he ascertained the drying-time by mixing the linseed oil with 5 per cent of siccative and expected it to dry in 24 hours. That this method is not free from objection can easily be seen, as it depends greatly on the quality of the siccative; one can therefore only obtain concordant results by using the same siccative, everything else remaining the same. Amsel states in a later paper that linseed oils which take 4—5 days to dry should not at once be rejected; in such cases, the iodine constant should also be ascertained, from which we may draw conclusions about the purity of the oil.

The influence of the temperature on the rapidity of drying has been shown by Weger[2]), who found that the drying of linseed oil took 5 to 7 days in the winter, 3 to 4 days in the summer, 12 hours at 50° C., 1 hour at 95° C., and only ½ hour at 120° C. when exposed in films.

The temperature at which the absorption of oxygen is to be determined, must be decided on. Suppose that the test was to be made at 10° C., then the maximum weight would occur after a much longer interval and would also be much smaller than at higher temperatures, because during the drying, a volatilisation takes place, which will be the greater, the longer before the maximum weight is reached. It is also important that the sample should be freely exposed to air. It is best to place the sample in a room where the temperature does not vary much, a room facing north is most suitable.

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[1]) Über Leinöl und Leinölfirnis, Zürich 1895.
[2]) Chem. Zentralblatt 1890, II, 127.

I have used Lippert's method with glass-plates, and have found for commercial linseed oils an increase of from 12·3 to 16·5 per cent. The samples were placed in a room which was not heated, and there dried in 6 to 8 days at a temperature of 8—10° C. The linseed oils expressed by myself reached their maximum weights in 5—6 days and they were between 15·0 and 18·2 per cent. I should add that the commercial oils were over one year old and the oils expressed by myself were quite fresh.

The absorption of oxygen by linseed oil goes on slowly and then suddenly reaches a maximum; then a rapid and afterwards gradual diminution in weight takes place.

Adulterations of linseed oils and especially serious additions of foreign oils can be detected by the specific gravity, the iodine constant, the drying-power and the absorption of oxygen. The following oils are used for mixing with linseed oil. Rape oil, cotton-seed oil, train oil, pumpkinseed oil, sesamé oil, rosin oil, mineral oil and sometimes also rosin.

Rape oil lowers the specific gravity as well as the saponification and iodine constants; besides this, rape oil contains sulphur and does not dry.

The presence of cottonseed oil can be shown by its qualitative reactions, it also lowers the iodine constant.

Train oil can be detected by its characteristic smell and also by means of colour reactions; it also diminishes the absorption of oxygen.

Pumpkinseed oil, which has been produced for some time in Hungary, is not used for mixing with linseed oil, but is employed in making boiled oil. Pumpkinseed oil has a smaller specific gravity, a smaller iodine constant and a considerably smaller refractometer-constant than linseed-oil. As it does not belong to the drying oils, its presence can also be detected by its absorption of oxygen.

Sesamé oil influences the constants of linseed oil in the same direction as pumpkinseed oil. One can always decide positively that an adulteration has taken place, but it is not always possible to ascertain the nature of the oils which have been added; if for instance rape oil or sesamé oil has been added, then it will be difficult to decide which of the two has been used.

The presence of rosin oils and mineral oils can be shown by the low saponification and iodine constants. Rosin oil increases the density, while mineral oils make the linseed oil less dense. As rosin oil and mineral oil cannot be saponified, their presence can be recognised in the unsaponified portion. Rosin oils can also be detected by polarisation, as shown by Aignau[1]) and Filsinger[2]).

The presence of rosin increases the very low acid-constant of linseed oil.

The method recommended by Amsel to show the presence of rosin oil and cottonseed oil by the thickening of the oil when mixed with zinc-white is according to W. Lippert[3]) absolutely valueless; because the cheesy thickening must be traced to the free fatty acids, and the rosin acids act less on the oxide of zinc than the fatty acids.

The table below contains the results obtained on examining various raw linseed oils.

Source	Specific gravity	Acid constant	Saponification constant	Iodine constant	Refractometer 40° C.	Absorption of oxygen
Győr	0·930	1·6	191	166	72·0	13·1
Győr	0·931	1·7	192	164	73·0	15·9
Oderberg	0·931	1·1	189	173	72·3	12·3
Triest	0·931	2·2	191	167	72·6	14·9
Lobositz	0·930	2·9	192	159	72·0	13·9
Holland	0·932	1·5	190	166	73·5	12·4
Győr	0·930	2·8	190	170	73·0	12·3
Győr	0·928	3·1	190	165	72·5	15·4
Triest	0·930	2·5	190	164	73·4	12·5
Lobositz	0·931	2·5	187	162	72·0	16·1
Breslau	0·931	2·9	188	162	72·4	15·7
Lobositz	0·930	2·0	190	164	72·1	15·1
Lobositz	0·931	2·4	194	164	72·1	15·2
Lobositz	0·931	2·3	191	165	72·0	16·5
Lobositz	0·931	2·3	189	163	72·8	14·6
Triest	0·932	1·3	187	171	72·8	14·9
Expressed by the author	0·933	0·2	192	177	73·7	16·1
	0·932	0·2	192	178	73·8	15·0
	0·931	0·7	190	178	73·5	16·9
	0·929	0·8	191	174	73·5	18·2

[1]) Chem. Zentralblatt 1890, II, 127.
[2]) Chemiker-Zeitung 18, 1005.
[3]) Zeitschrift f. angewandte Chemie 1897, 779.

It is evident that the data given in the above table correspond to those indicating pure linseed oils. The iodine constants are considerably lower than many authorities maintain; but even the iodine constants of oils expressed by myself do not reach 180, as required by Rowland Williams. As a greater excess of iodine was used as well as a longer time of action, the lower iodine constant can only be ascribed to the source of the oil. That the absorption of oxygen is lower than Weger has found it to be, may be explained by the lower temperature at which the tests were made.

Boiled linseed oil.

Boiled oil is produced by heating linseed oil with substances, so called siccatives, which give up oxygen readily. The product thus obtained should dry within 24 hours when exposed to air in thin films. It is now required that the drying time of boiled oil should not exceed 12 hours; there are even boiled oils which become hard in 5—6 hours.

Amsel distinguishes between high and low temperature oils, including among the latter such drying oils as are produced by treatment with a siccative at 150° C. or even at lower temperatures. Boiled oils were formerly made by letting linseed oil boil for some time at 220°—300° C. adding at the same time some manganese dioxide, litharge, red lead, or borate of manganese. When applying these oxidising substances, except the borate of manganese, the above named boiling temperatures are necessary; but good drying oils can be obtained at 200° C. or even at 160°—170° C., if air be blown through the heated mass. Boiled oils prepared with the aid of special soluble driers only require heating to 130°—150° C.; there are even liquid preparations by the use of which the linseed oil need only be heated to 60°—70° C., provided air is blown through the mixture. The special driers are linoleates or resinates, i.e. lead or manganese soaps of linoleic or resin acids.

In order to test a boiled oil for purity, the following determinations must be made:

1. Specific Gravity. Ulzer[1]) gives the specific gravity of boiled oil as between 0·935 and 0·940; this is however not correct. I have convinced myself by tests that the gravity can be as high as 0·960, when the oil has been boiled too long.

The specific gravity of Russian boiled oil is stated by K. Charitschkoff[2]) to be 0·926—0·9584, he says however that as a rule it is 0·939—0·940. It is difficult to understand how the specific gravity can be lower than that of raw oil which is at least 0·928. The only explanation for this is that the material has been prepared by a cold process, without any heating, and in presence of a drier containing turpentine. I have found the specific gravity of boiled oils I have tested to be 0·937—0·955. A higher specific gravity indicates that the oil has been boiled too long, or the air-blast has been kept on too long during boiling or afterwards, but in either case while in a warm state.

2. Iodine Constant. The iodine constant of boiled oil is smaller than that of raw linseed oil, and the smaller it is, the longer was the oil heated and the higher was the temperature at which boiling was done. The oxidation also lowers the iodine constant: old boiled oil shows a smaller iodine constant than a freshly made sample.

Hefelmann[3]) has tested pure commercial boiled oils and found iodine constants between 163 and 172. Fahrion[4]) found the iodine constant of a boiled oil which had been kept in an open bottle for a year to be 151. Boiled oils which have been made with soluble driers give higher iodine constants than those which have been heated with oxidising substances at high temperatures. The iodine value is also lowered when a large quantity of resinates have been introduced into the oil. As the iodine constant depends on the process by which the boiled oil has been made, its determination does not teach much, for no conclusion can be drawn from it alone unless it is abnormally low. Pure commercial oils which have been boiled with resinates of lead and manganese, according to my tests, give iodine constants from 153 to 167: these oils

[1]) Analyse der Fette und Wachsarten, R. Benedikt, 1903, 591.
[2]) Chem. Revue der Fett- u. Harzindustr., 7., 1.
[3]) Pharmaceut. Zentralbl. 1895, 685.
[4]) Zeitschrift für angew. Chemie 1892, 6.

were not tested till 6 months after they were made and they had been kept hermetically sealed all the time.

3. Acid Constant. I have shown how small the acid constant of raw linseed oil is. On the contrary, the acid constants of boiled oils are much higher, and according to Ulzer may reach 12.

The acid constant is increased by boiling the oil for a long time, and also when the resinates, which are used for boiling, contain free rosin. Boiled oils tested by me have given acid constants between 6·6 and 15·6.

4. Saponification Constant. The saponification constant of boiled oil is similar to that of raw linseed oil; if therefore the boiled oil is found to have a saponification constant lower than 188, it is certainly adulterated. Amsel[1]) has by means of the already mentioned water reaction found very high values for the saponification constant. He considers that the saponification constant of boiled oils made from pure linseed, should be between 190 and 195, and with smaller saponification constants must be considered as containing rosin oil or mineral oil. In this manner it should not only be possible to detect the presence of rosin or metallic resinates, but the amounts added should even be capable of approximate estimation. An oil for which he found a saponification value of 187 was reported by Amsel as having had 4 per cent of metallic resinate added; but the manufacturers stated that only 2·5 per cent of resinate had been used in boiling.

The water reaction is performed by dissolving 30 gms. of purest potassium hydroxide in 50 ccm of water and then diluting to 1 litre with 96 per cent alcohol. With this solution, the saponification will be finished in 20—25 minutes. After saponification, the alcoholic soap solution is diluted with water, and it is observed whether the solution becomes milky.

The incorrectness of the conclusion which Amsel arrived at is proved by the following test made by himself: Under No. 19 in the table which appears in his paper, is cited a boiled oil which has a saponification constant of 191 and an iodine constant of 125. The oil is represented as having been adulterated with 50—60 per cent of rape oil. It is quite true that the low iodine constant

[1]) Zeitschrift für angew. Chemie 1895, 73, 262.

points to an original oil with low iodine constant, but as the saponification constant is 191, it is impossible that there can be 50 per cent rape oil present. If we take the saponification constants of pure boiled oil and of rape oil at their highest, say as 195 and 179 respectively, then the saponification constant when 50 per cent rape seed oil is added, could not be higher than 187. Likewise if we take the iodine constant of boiled oil as 165, and that of rape oil as 99, then the iodine constant when 50 per cent of rape oil is added must be 134, and will not be 125 until 60 per cent of rape oil has been added.

As already mentioned, Lippert has shown that the water reaction is untrustworthy; if therefore a boiled oil has a low saponification constant, it only remains to test its unsaponified portion. Large quantities of rosin oil can be estimated by determining the amount of matter soluble in alcohol. As Lippert[1]) has proved, the water reaction can also be obtained when there are no unsaponified substances present. Lead and lead resinate oils do not give a water reaction; but it is given by oils which have been produced with manganese preparations. This is a matter of course, as in the case of a lead oil, the separated lead hydroxide will be held in solution in the excess of alkali, and the alkali resinate which is formed is soluble in water. In the case of oils which are made with manganese resinate or other manganese combinations, a turbidity is produced in the alcoholic soap solution by adding water; but this turbidity is caused by the separated manganese hydroxide and not by the resin of the resinate.

Rosin, rosin oil and even mineral oil can be present in boiled oil without the water reaction occurring; a turbidity can therefore take place, without the oil being adulterated. Hence, if the water reaction produces a turbidity, which is not due to manganese hydroxide, one can conclude that there is a considerable amount of mineral oil or rosin oil present.

I have found the saponification constants of pure commercial boiled oils to be between 187 and 198; the lower limit is therefore somewhat below that given by Amsel, and agrees with that of raw linseed oil, which is even higher.

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[1]) Zeitschrift für angew. Chemie 1897, 655.

5. Refraction. The refractometer constant of boiled oil is higher than that of raw linseed, whose refractometer constant increases by the treatment or on keeping. The refractometer constant of boiled oil increases by adding oxygen, by blowing air through it or on keeping. The refractometer constant of linseed oil is thus increased by boiling with oxidising substances.

Weger found the refractometer constant of a linseed oil to be 80·2 at 25° C. and to be increased to 86·7 by heating it for one hour to 150° C. in presence of 5 per cent of manganolead resinate. When there is no adulteration, a high refractometer constant points to a highly heated or a blown oil, while a low refractometer constant indicates an oil heated to a low temperature with soluble driers.

The refractometer constants of the boiled oils examined by the author were between 73·8 and 78·5.

6. Absorption of Oxygen. The absorption of oxygen by a boiled oil is found by the Lippert plate method in the same manner as explained under linseed oil. As the boiled oil already reaches its maximum absorption in 12 hours, the test sample must be spread out in the evening and weighed again in the morning. Weger found the oxygen-absorption of boiled oils prepared in different ways to lie between 14·6 and 17·6.

The oxygen-absorptions of the boiled oils prepared with mangano-lead resinate which I have examined were between 11·4 and 14·3; the samples being 9 months old when tested, and having been submitted to an air blast while boiling.

One cannot conclude much from the absorption of oxygen; as Lippert[1]) has shown, boiled oils adulterated with rosin oil can absorb a greater amount of oxygen than when they are genuine. According to his tests it appears that rosin oil alone does not show an increase in weight but a diminution in weight. Rosin oil in presence of siccatives absorbs oxygen and can thus reach a higher weight-maximum than boiled linseed oil.

If the absorption of oxygen is high, then the oil has been prepared with small quantities of siccative at a low temperature

[1]) Zeitschrift für angew. Chemie 1899, 513, 540.

and without an air blast. If the oil is undoubtedly genuine, and the oxygen-absorption is low, the boiled oil has been prepared with large quantities of driers and a powerful air-blast; or it has been raised to a high temperature. If, however, the boiled oil has been adulterated with substances which absorb very little or no oxygen, the oxygen constant will be low; whereas pure boiled linseed oil has a high oxygen-absorption.

The plate-method by itself is not decisive for judging a boiled oil. It is however of some importance if one notes the time taken for the oxygen-absorption to reach its maximum, and then observes whether the surface is still sticky. In the presence of even 5 per cent of rosin or of rosin oil, the absorption of oxygen may be normal, but the oil will still remain sticky.

7. Drying-Time. According to Amsel, a boiled oil requires 24 hours for drying; the quantity of oil required for this test is as small as that used for the plate-method. The oil must be excluded from direct sunlight and the temperature of the room must be even. Several factors influence the drying-time such as temperature, light, dampness and the quantity of siccative with which the oil was boiled.

In testing a boiled oil for drying, it is not only necessary to observe the time it takes to dry, but also the manner in which the drying takes place, especially whether the film does not remain sticky. The origin of the linseed oil has some influence on the hardness of the film; for instance the film from Russian linseed oil is harder than that from Indian oil, notwithstanding similar treatment. Air-blown oil does not dry so hard as unblown. An oil to which rosin has been added, dries badly; it remains sticky. If however the rosin is hardened with lime, then it will produce a hard film; but this can easily be detected, as a drop of water placed on the dry skin will produce an intensely white spot. This spot will also be produced when manganese dioxide containing a large amount of lime has been employed.

I have compiled the results of my tests in the table below. All the samples were prepared with mixed resinates of lead and manganese.

No.	Specific gravity	Acid constant	Saponification constant	Iodine constant	Refractometer 40° C.	Absorption of oxygen
1	0·941	11·8	188	154	75·5	12·2
2	0·941	11·0	191	153	78·0	13·4
3	0·942	11·1	196	155	76·1	12·7
4	0·951	13·4	197	157	77·0	11·4
5	0·948	9·2	198	155	78·3	12·5
6	0·944	10·2	187	159	76·8	12·2
7	0·948	10·5	189	161	78·5	12·5
8	0·938	4·8	189	168	74·1	12·4
10	0·945	6·7	188	167	76·1	12·8
11	0·949	6·6	192	167	75·0	13·8
12	0·937	9·5	197	159	74·5	14·3
13	0·955	15·6	194	153	74·2	11·9
14	0·941	10·4	194	154	73·8	11·5
15	0·941	10·9	189	154	73·8	11·4

Rosin oil comes first on the list of substances which are used for adulteration; it is however easily detected by its low saponification and iodine constants, by polarisation, and by the dry boiled oil still remaining tacky.

Filsinger[1]) in his polarisation tests mixes 1 volume of boiled oil with 1 volume of alcohol and 2 volumes of chloroform. Pure rosin oil produces a rotation through 25°—30° to the right. The sample submitted to a polarisation test must not contain any turpentine, the presence of which will be shown by the oil losing weight when spread out in a thin layer. Pure boiled oil causes hardly any deviation of the plane of polarisation; an oil made with resinates produces a very small deviation.

When rosin oil is absent, the presence of mineral oils will be shown by the lower saponification constant and by examining the unsaponified portion. If both are present, then the rosin oil can easily be found in the unsaponified portion by the Storch-Morawsky or the Holde reaction, and it can be separated from the mineral oil by the Storch[2]) method. A rosin oil will mix with

[1]) Chem.-Zeitung 1894, 1867.

[2]) Berichte der österreichischen Gesellschaft zur Förderung der chemischen Industrie 1887, 93.

acetone in any proportion, while mineral oils are only soluble in several times their own volume, therefore this method can be applied to separate both.

An oil to which mineral oil has been added, does not dry. With regard to the determination of the unsaponified portion, it must be observed that boiled oils contain about 1 per cent of unsaponified matter, which will be increased by resinates.

If a boiled oil contains rosin, the latter will diminish the iodine constant as well as the saponification constant, while the acid constant will be higher. Rosin also makes the film sticky unless it is hardened with lime. The presence of rosin can also be shown by the Storch-Morawsky process in the following manner: The oil is to be shaken with absolute alcohol, and after the mixture has been left standing for some time, the alcohol is evaporated. The residue is dissolved in acetic anhydride to which is then added sulphuric acid having a specific gravity of 1.53. If rosin is present, the sulphuric acid will become red, while at the place of contact the acetic anhydride will become violet red; the same takes place in the case of rosin oil. It is thus possible to show that the oil has been boiled with resinates; but it must be noticed that this indication of the presence of resinates does not always succeed. Some oils give with this reaction a brown-red colouring, which may hide the red and violet-red colouring; if notwithstanding this, the reaction is obtained, then there is either rosin present or the oil has been prepared with resinates.

Train oil is also used for adulterating boiled oil and chiefly Menhaden-train oil. Its presence can be shown by Salkowski's[1]) cholesterin test and its modifications by Bömer[2]) and Marcusson[3]).

Boiled oil adulterated with train oil absorbs less oxygen, which makes it possible to indicate its presence; neither the iodine constant nor the saponification constant is influenced by Menhaden-train oil. Train oil can also be recognised by the characteristic colour-reactions which are obtained by sulphuric acid and phosphoric acid. Thus sulphuric acid of specific gravity 1·53 produces a red

[1]) Zeitschrift für anal. Chemie, 26, 565.

[2]) Zeitschrift für Untersuchung der Nahrungs- und Genußmittel 1898, 21.

[3]) Mitteilungen der Versuchsanstalt Berlin, 1900, 261; 1901, 259.

to purple-red colouring, according to the nature of the train oil. This reaction is however only applicable when rosin oil is not present, as the latter gives the same reaction. Syrupy phosphoric acid produces with train oil a similar red colouring, which is very characteristic. Train oil may also be recognised by its smell.

Chinese wood oil has been used of late as an addition to boiled oil and especially when a quick drying and hard material is required. This ingredient cannot be considered as an adulteration as it is more expensive than linseed oil. As the melting point of the fatty acids contained in boiled linseed oil is 20—21° C., whereas the fatty acids of wood oil melt at 43—44° C., the presence of Chinese wood oil can be detected in that way.

If it be required to determine which metals are contained in the oil, we must either examine the ash or employ the method due to A. Fresenius and A. Schattenfroh, by which the oil is shaken together with nitric acid, and the acid is tested for metals, of which chiefly manganese and lead will be found. Large quantities of lime point to resinate of lime.

The preceding investigations show that the purity of a raw or boiled linseed oil cannot be ascertained by determining the saponification constant, water reaction, drying power or the portion soluble in alcohol; for even when these results point to the sample being genuine, an adulterant may yet be present. It is therefore necessary to carry out all the tests by which the absence or presence of foreign matter can be detected.

A. Grittner.

Budapest, December 1903.

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22 e.

INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 22.

Group C

REPORT ON THE WORK OF COMMITTEE 22. APPOINTED FOR THE SOLUTION OF THE PROBLEM „UNIFICATION OF TESTING METHODS“

PRESENTED BY THE CHAIRMAN OF
COMMISSION 22
PROFESSOR N. BELELUBSKY.
ST.-PETERSBURG.

(TRANSLATED BY F. H LEEDS, F. I. C.
LONDON.)

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Report on the work of Commission 22

presented by **Professor N. Belelubsky**, St.-Petersburg, Chairman of Committee 22.

(Translated by F. H. Leeds, F. I. C., London.)

The text of problem 22 which was proposed at the Zurich Congress 1905 reads as follows:

Considering that the resolutions formed by the International Conferences of Munich, Dresden, Berlin, Vienna and Zurich for the purpose of attaining unity in the methods of testing materials and the report of the Committee of the Am. Soc. Mech. Eng. do not agree in many points with the decisions arrived at by the French Commission, it is proposed: that the Council appoint a commission which shall prepare a report upon these differences, and proposals for ways and means of abolishing them. (Proposed at the Zurich Congress 1895.)

At the Budapest Congress 1901, the Commission appointed by the Council for the solution of this Problem consisted of following members:

Chairman:

Belelubsky, N., professeur, directeur du laboratoire méc. à l'Institut impérial des ing. des voies de comm., membre du conseil du ministère; St. Pétersbourg.

Vice-Chairmen:

Martens, A., Geheimer Regierungsrat, Professor, Dr. Ing. Direktor des kgl. Materialprüfungsamtes in Groß-Lichterfelde, W.

Sauvage, Ed., professeur à l'Ecole des mines, ingénieur en chef des mines et de la Cie de l'Ouest; Paris.

Members:

Belgium: Camerman, E., ingénieur-chimiste des chemins de fer de l'Etat belge; Bruxelles.

Roussel, E., ingénieur, chef du laboratoire d'essai à l'arsenal de Malines.

Denmark: Hannover, H. I., Professor der kgl. technischen Hochschule; Kopenhagen.

Germany: Borries, von, Geheimer Regierungsrat, Professor der technischen Hochschule Charlottenburg; Berlin, W.

Dyckerhoff, R., Fabrikbesitzer, Portland-Zementfabrik Dyckerhoff & Söhne; Amöneburg bei Biebrich am Rh.

Eger, Regierungs- und Baurat, Ministerium der öffentlichen Arbeiten; Berlin.

Gary, M., Ingenieur, Vorsteher der Abteilung für Baumaterialprüfung des k. Materialprüfungsamtes in Groß-Lichterfelde, W.

Krohn, R., Prof., Geh. Regierungsrat; Danzig.

France: Baclé, L., ingénieur; Paris.

Candlot, E., fabricant de ciment Portland; Paris.

Feret, R., chef du laboratoire des ponts et chaussées; Boulogne-sur-mer.

Mesnager, A., professeur à l'Ecole des ponts et chaussées, ingénieur des ponts et chaussées; Paris.

Hollande: Alpherts, G., Vorstand des technischen Bureaus im holländ. Kolonialministerium; Haag.

Bienfait, L., Ingenieur, Prüfungsanstalt für Baumaterialien; Amsterdam.

Italy: Biadego, B. G., ingegnere, direttore delle costruzioni delle Ferrovie Mediterranee; Roma, via Mercede 9.

Mattei, V. de, direttore di Fabrica de Calce e Cementi Casale, Monferrato.

Austria: Berger, Fr., k. k. Oberbaurat, Stadtbaudirektor; Wien.
Dormus, A. R. v., Oberingenieur der Kaiser Ferd.-Nordbahn; Wien.
Kirsch, B., Professor der k. k. techn. Hochschule; Wien.
Tetmajer, L. v., k. k. Hofrat, Professor, Wien (†).

Russia: Antochine, N., ingénieur, membre du conseil du génie au ministère des voies de comm. St. Pétersbourg.
Baykoff, A., professeur à l'Institut polyt, St. Pétersbourg.
Bogouslawsky, N., agent supérieur, ingénieur d'administration des chemins de fer de l'Etat russe; St. Pétersbourg Razjezjaïa 13.
Gagarine, prince, directeur de l'Institut polyt., St. Pétersbourg.
Konosiewicz, ingénieur; St. Pétersbourg, Zabalkansky 9.
Taguéeff, Gr., ing. directeur des travaux du Port à Enzeli.

Sweden: Wahlberg, A., Chief Engineer of the Fagersta Works; Fagersta.
Berg R. F., Ingenieur, Direktor der Skanska Cement Aktie Bolaget, Malmö.

Switzerland: Schüle, F., Professor am eidg. Polytechnikum; Zürich.

Hungary: Nagy, D. von, Professor der kgl. ung. technischen Hochschule; Budapest.

United States: Colby, Albert Lad., International Nickel Comp. 43 Exchange Pl, New-York.
Henning, Gus. C., Consulting Engineer; 521 W 142 str., New.-York.
Moldenke, R. G., Dr., Metallurgist and Consulting Engineer; Box 432, New-York City, N. Y.
Webster, William R., Civil Engineer; 411 Walnut Street, Philadelphia, Pa.
Wood, Walter, Cast Iron Pipe Manufacturer R. D. Wood Co.; 400 Chestnut Street, Philadelphia Pa.
Swain George, F., Professor of Civil Eng. Massachusetts Inst. of Technology; Boston, Massachusetts.
Webster George, S., Chief Engineer and Surveyor; Bureau of Surveys, 318 City Hall, Philadephia Pa.

All technical men who have to do with materials that are used in building and construction should feel great interest in a brief review of the methods which have met with general acceptance for testing them, because such an inquiry will show to what a large extent the present position of this particular branch of technology is due to theoretical and practical study co-operative in its nature.

Twenty-five years ago, before uniformity was introduced, the testing of different materials employed in building and machine construction had reached a high state of development, being chiefly directed to the formulation of technical rules governing the supply and acceptance of such articles. Even at the time in question many classical investigations had been published which might be instanced as foreshadowing the advent of standard methods. Hence conditions were so far favourable that the notion met with general approval when, at the suggestion of Dr. Michaëlis of Berlin and Professor v. Tetmajer of Zurich, international conferences for the settlement of standard methods of testing materials were instituted in 1884 by the late Professor Bauschinger of Munich, whose death we all have reason to deplore. The two first conferences, held at Munich in 1884 and at Dresden in 1886, prepared the way for the introduction of uniform methods for testing the most important materials, methods which were again examined and amplified at the next two conferences held at Berlin 1890 and Vienna in 1893, also under the Presidency of Professor Bauschinger. Technical men were present at all these conferences belonging to each of the nations participating in them, and the results obtained in different laboratories, as well as the researches carried out by certain specialists, contributed largely to the best possible solution of the problems under consideration. At the request of the Berlin meeting, Bauschinger published the resolutions passed by the four conferences in the "Proceedings" of the laboratory of the technical High-School at Munich. The shorthand report of the conferences contains an extremely interesting collection of the most important investigations, the reports of the sub-committees, and the discussions upon the resolutions brought before the different conferences.

Even from their very inception these early conferences were international in character. Those of us who took part in them still remember the thoroughly impartial attitude of Prof. Bauschinger

in the Chair; and how that attitude has caused the resolutions which were passed, purely optional though they were, to meet with general recognition, and to exert a marked influence upon methods of testing, and upon the conditions governing the acceptance of materials. Through the introduction of standard methods, the results obtained on testing materials have become comparable, and so they lead to further improvements and additions to the methods proposed.

The fifth International Conference was held at Zurich in 1895, under the Presidency of Professor v. Tetmajer, who succeeded to the Chair rendered vacant by the death of Professor Bauschinger. This meeting must be regarded as a true International Congress, for, thanks to the energy displayed by the President, several countries such as France, Great Britain, Italy, and the United States, which had been but partially represented on former occasions, participated largely in the conference.

The French visitors, who were mostly members of the French national Commission for revising the methods of testing employed in that country, and who included in their numbers MM. Quinette de Rochemont, Polonceau, Osmond, and Debray, took the opportunity of presenting to the Congress the Transactions of the French Commission, contained in four bulky and interesting volumes.

Moreover, during the French International Exhibition of 1889, two congresses were held in Paris upon building methods and mechanics, at which questions relating to uniformity in testing and to technical specifications for iron and steel were much discussed.[1]) As a consequence of these congresses, an official Commission for testing building materials (Commission des Méthodes d'Essai des Matériaux de Construction) was appointed in France during the year 1889, an account of which was given by Professor Debray to the St. Petersburg meeting of the Railway Congress in 1892.

The members of the French Commission were nominated by the Government, and comprised many men of great scientific activity. The four volumes of Transactions, published at the expense of the French Government, bear witness how the labours of the Commission

[1]) The writer of the present report was enabled to read a paper in Paris describing the conclusions arrived at by the Dresden conference with respect to tests of building materials for their ability to resist frost, and upon the manner in which such tests were carried out in Russia.

have enriched every department of material testing. On its own part the Commission has done much work, based on independent researches performed by French investigators, which brings into prominence special points connected with every single branch of testing. Although some French engineers have attended the International Conferences held since 1890[1]) and have become members of the standing committees appointed by them, the French national Commission has steadily carried on its work, up to the date of the Zurich Congress, without any reference to the labours of the International Conferences.[2])

The foundation of an International Association for Testing Materials used in the Arts during the year 1895 must be regarded as a natural consequence of the large international attendance at the Zurich Congress. The fourth committee appointed at the Zurich meeting was entrusted with the constitution of the Association and the drafting of its By-laws by our lately deceased President Professor v. Tetmajer, to whose tireless exertions the International Association mainly owes its very existence.

One of the primary objects of the society was to compare the opinions expressed by single committees in different countries with the resolutions passed at the international Conferences, to submit the experimental results and the observations of members from each nation to a general discussion, and so to hasten the introduction of standard methods of testing which were so urgently required for both practical and scientific reasons. Hence, by a resolution passed on April 26th 1896, the Council of the newly-formed International Association appointed an international committee to deal with this matter, M. Polonceau of Paris being the Chairman, and Professor Martens of Berlin being nominated Deputy-Chairman. The Terms of Reference to the committee stated that:

> The resolutions passed by the International Conferences at Munich, Dresden, Berlin, Vienna, and Zurich relating to the introduction of standard methods for testing constructional

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[1]) Professor Debray and M. Candlot were present at the Berlin conference (1890) in their official capacities.

[2]) It should be remarked that the permanent committees have always kept French methods of testing in view, although French engineers did not take part in the first Conferences. For example, the Dresden Conference in 1886 adopted the French apparatus for testing ballasting materials as the standard machine.

materials exhibit considerable differences from the conclusions arrived at by the "Commission Française des Méthodes d'Essai des Matériaux de Construction" and from the opinions expressed by the American Society of Mechanical Engineers. The Council of the International Association for Testing Materials has therefore appointed a committee to prepare a report upon the differences and upon the manner in which they may best be removed.

The proceedings of the committee.

First period, 1896—1897.

The Polonceau-Martens committee numbered about 40 members residing in all parts of the world, and during the first year of its existence, the committee was engaged in formulating general schemes of procedure for submission to the individual members. For instance, at the request of the Chairman, draft processes for the examination of metals and hydraulic cements were kindly prepared by MM. Baclé and Alexandre of Paris, members of the committee. These two drafts were distributed amongst the members, who were invited to express their views on them: the remarks made being in their turn circulated among the members of the committee, until at length it became possible to prepare a joint report from the material collected. M. Baclé's draft scheme for the examination of metals included physical and chemical tests, general advice, experiments with static and impact testing machines, tests of hardness and brittleness, and a description of some experiments upon specially prepared test pieces.

Observations upon both draft testing processes were received from the following members: MM. Alpherts, Belelubsky, Berger, Biadego, Bienfait, Borries, Candlot, Dyckerhoff, Eger, Hannover, Henning, Krohn and Martens.

As will be easily imagined, the magnitude and importance of the task, and its late imposition upon the committee, prevented a complete account of the different criticisms passed upon the two drafts from being prepared in time for submission to the Stockholm Congress. Nevertheless, an interim report upon the opinions expressed by the members was drawn up by M. Polonceau, the Chairman, was approved by the committee at a meeting held

shortly before the Congress met, and was presented to the Congress at Stockholm. This report showed how necessary it was to study the numerous weighty observations made by the members of the committee, although, broadly speaking, they agreed with the recommendations originally offered by the French authors of the drafts. Moreover, a special note was handed in by the German members of the committe before the Congress met, expressing the opinion that the principles guiding its future labours should be laid down before the real work of the Stockholm Congress was entered upon. Thoroughly appreciating the value of the suggestions made by their French colleagues, the German members dealt at some length with the main points that should be considered in any international resolulions. They also discussed the question as to the standpoints from which international resolutions ought to be drawn up.

As the Congress took for granted that the time at the Commitee's disposal would not be sufficient to allow of a final report being presented, the Stockholm meeting decided that „The international Committee should be reappointed for another period without any alteration in its terms of Reference." It may be added that in conformity with the President's desire, and the wish expressed by many members, it was determined to keep the resolutions of the Bauschinger's Conferences in mind as far as possible during the future proceedings of the Polonceau-Martens committee.

The proceedings of the committee.

Second period, 1897—1900.

Following the resolutions passed by the Stockholm Congress, a number of committees were appointed by the Council of the Association to meet until the next Congress (Paris, 1900) for the discussion of technical problems. A complete list of these committees was published in the form of a pamphlet which also contained rules, lists of the members, and the various agenda. The committee appointed to consider the differences in existing methods of testing (as already mentioned) was designated Committe No. 22.

Owing to the resignation of M. Polonceau, the Chairman, Professor Debray, member of Council, was appointed to the post, M. Polonceau being nominated Honorary Chairman.

The committee desires to place on record its sense of the highly valued services rendered by its late Chairman.

In February 1899 was prepared under the supervision of Professor Debray, of Paris, a draft scheme, including all the resolutions passed relating to the examination of metals and cementitious materials, and also as far as possible incorporating the suggestions made, at the request of the former Chairman, by the members.

It appeared that in altering the general arrangement of the preliminary scheme in accordance with the wishes expressed by the German members certain difficulties would arise, but that the changes already embodied in the resolutions nevertheless agreed with the proposals of the Conferences presided over by Bauschinger.

In February 1899 the German members of the international committee Nr. 22 laid before their fellow members their proposals for the alteration of the resolutions passed by the International Conferences dealing with the introduction of standard testing methods. The proposals set forth in parallel colums: *a*) the early recommendations of the Conferences (viz. the Bauschinger Conferences, and the meetings at Zurich and Stockholm); *b*) the new proposals of the German committee-men; *c*) the later proposals of the members of committee No. 22 based upon the various counter proposals emanating from discussions of the original drafts prepared by Baclé and Alexandre.

The proposals of the German members related at first to the testing of metals only, as it had been originally agreed to accept M. Alexandre's draft without sensible alteration. The joint proposal arising out of the various criticisms was submitted by the committee to discussion at a meeting held in Paris on February 10th by Professor Debray dealt chiefly with the two drafts prepared by MM. Baclé and Alexandre and with the comments made upon them.

In order to coordinate the proposals of the German members, the numerous remarks handed in, and all the investigations already carried out, the committee appointed two sub-committees at its Paris meeting. Sub-committee „A", consisting of MM. H. Hannover of Copenhagen, E. Roussel of Mechlin, and G. Alpherts of The Hague, was instructed to consider the mechanical examination of metals and alloys, while sub-committee „B", consisting of MM. L. Bienfait of Holland, E. Candlot of Paris, and M. Gary of Berlin, was entrusted with the testing of hydraulic cements.

The members of sub-committees „A“ and „B“ under the guidance of the Chairman, Professor Debray, and the Vice-Chairman, Professor Martens, took the opportunity of the Council meeting which was held in Dresden, September 1899, to express their opinions on certain points after Professor Hannover had publicly made a provisional survey of the various recommendations.

The work of sub-committee „A“ falls into three divisions: the first contains the recommendations of the sub-committee; the second contains the sources of their observations, and the reasons for the changes which they felt it necessary to make; and the third contains miscellaneous comments. When the different recommendations are not in agreement, the fact is mentioned in the preamble of the report, and the points in dispute are discussed. Sub-committee „A“ also devotes special attention to the resolutions passed at the early Conferences (the Bauschinger-Conferences, or „K. B.“), the resolutions of the French national Commission („F. D.“), and the resolutions of the German Committee („D.“).

The report of sub-committee „A“ contains the resolutions of the early Conferences with respect to methods of performing mechanical examinations (experiments upon ultimate strength and technological tests); and as an appendix describes the application of these methods to malleable and cast iron, to copper, and to other metals and alloys.

The report of sub-committee „B“ contains some brief recommendations with respect to the use of those standard test methods which are most commonly employed, and for which standardisation is most requisite, other matters being postponed to be dealt with later.

The reports of the two sub-committees have been printed in both the German and the French languages and distributed amongst the members of the parent committee.

It was found impossible to lay before the Congress upon Methods of Testing, which met at Paris during the International Exhibition, 1900, the report of committee No. 22, drawn up at the request of the International Association for Testing Materials; because the Paris Congress, not having been formally constituted by the Association, was not qualified to receive a report unless it had been previously atopted by the committee and by the Council of the said Association,

In the autumn of 1900, Professor Debray was compelled, owing to business reasons, to resign his membership and Chairmanship of committee No. 22, together with his seat on the Council of the Association, so that the latter body had to face the difficulty of reorganising the committee in question. After M. H. Le Chatelier had accepted the invitation of the Council to become a member, the VIII[th] meeting of Council held in February 1901 proceeded to the election of a Chairman for committee No. 22; and since Professor Tetmajer, Professor Martens, and Professor Le Chatelier declined the honour, the author of the present report was constrained to accept the post. Recognising the onerous nature of the duties imposed upon him, the writer could only rely on the friendly assistance of the Vice-Chairmen, Professor Martens of Berlin, and the newly-elected Professor Sauvage of Paris. At the same meeting, Professor Mesnager of Paris was elected a member of the committee in place of Professor Debray. For the composition of committee No. 22 at this time, reference may be made to the beginning of the present report.

The proceedings of the committee.

Third period, 1901.

Before the reporter felt himself competent to take over the direction of the committee and to elaborate a plan for further work, he recognised the necessity of making himself acquainted with what had been done during the past two years, in pursuance of which he was fortunate enough to secure personal interviews with Professor Martens of Berlin, and Professor Sauvage, and the reporter Le Chatelier of Paris from whom he received most valuable advice.

The shortness of the time[1]) and the imminence of the Budapest Congress prevented a larger measure of activity being displayed, and therefore the committee, with the approval of the Council, was only able to bring before Congress the reports of the two sub-committees together with the remarks made by the members of the committee thereupon, and to secure their acceptance.

[1]) By official command, he was sent to Transcaspian Territory to take charge of the bridge over the river Amu-Darja and was therefore unfortunately hindered for about two months from devoting any time to the concerns of committee No. 22.

In April of the Congressional year, Professor Martens handed me a copy of the "Principles of Standard Methods of Testing" drawn up during 1900, with the suggestion[1]) that these principles be kept in mind when the standard methods for testing materials should be finally drawn up. Copies of this pamphlet in the German language were supplied to all the members of the committee.

As may be seen from the preface, the proposals of the German Association agree as thoroughly as possible with the recommendations of the earlier Conferences presided over by Professor Bauschinger and with the conclusions arrived at from work done in other countries, e. g. by the French «Commission des Méthodes d'Essai des Matériaux de Construction,« by the International Association, and by the American Society of Mechanical Engineers. These principles relate only to the mechanical testing of metals.

A circular letter was then sent to the members of the committee, requesting them to express their opinions upon the proposals of the two sub-committees. The French members, including Professor Le Chatelier and Professor Sauvage, considered that there was nothing to object to in the scheme of sub-committee «B» for the testing of hydraulic cements, and they accepted the scheme of sub-committee «A» relating to metals in its entirety but recommended to include the tests upon notched bars.

At a meeting of committee No 22 held in Budapest shortly before the opening of the Budapest Congress, the members were given an opportunity of expressing their opinions upon the proposals of the two sub-committees, and it was decided that these opinions should be taken as the basis of future investigation.

From this brief review will be seen that although the elaboration of the most important propositions urged by committee No 22 was beset by manifold difficulties, a considerable amount of progress had been made by the time of the Budapest Congress; and it might confidently be anticipated that the combined efforts of the members, with the assistance of the documents prepared by

[1]) During its meeting at Carlsruhe in 1896, the German Association appointed a committee to compare the resolutions relating to standard methods of testing passed by the early Conferences (held since 1884) with those passed by foreign Associations, and to make recommendations for the alteration or completion thereof.

the sub-committees (as already stated), aided by the labours of the German Association, of French and of American men of science, must eventually succeed in bringing the matters dealt with by committee Nr. 22 to a succesful issue.

At the Budapest Congress itself, the reporter as Chairman of committe Nr. 22, laid upon the table a short historical review of the work done by the committee, accompanied by the printed reports of sub-committees "A" and "B", the former of which related to metals and the latter to hydraulic cements. Adopting the committee's proposals, therefore, the Budapest Congress unanimously passed the annexed resolutions,[1]) to wit:

1. That sincere thanks be offered to the following authors for their comprehensive and valuable services in connection with the preliminary labors and their proposals with reference to unification: Mr. P. Alexandre and Mr. L. Baclé, of Paris, Mr. F. Alpherto, of the Hague, Mr. H. J. Hannover, of Copenhagen, Mr. E. Roussel, of Malines, Members of Sub-commitee A, and to Mr. L. Bienfait, of Amsterdam, Mr. E. Candlot, of Paris, Mr. Gary of Berlin, Members of Sub-commitee B.

2. That the Congress consider the collective propositions of the two Sub-committees and authorize the Committee to continue this study using as a basis the plans of the two Sub-committees, and with due regard also to the propositions of the German Association for Testing Materials, the results obtained by French and American Engineers, as well as the resulutions and suggestions offered at the present Congress.

3. That the Congress express its earnest desire that a final solution of problem 22 for the more important materials be reached in time for the next Congress.[2])

The proceedings of the committee.

Fourth period 1901—1906.

During the sittings of the Budapest Congress, an invitation was received from Russia to hold the fourth international Congress at St. Petersburg, and was accepted by the members with much

[1]) At the meeting of committee No 22 held in Budapest on September 8th 1901, the following gentlemen were present MM. Tetmajer, Belelubsky, Martens, Gary, Sauvage, Bienfait, Wahlberg, Baclé, Hannover, Eger and Nagy.

[2]) Compare the Minutes of the Budapest Congress, September 9th to 14th 1901.

pleasure. The members of Council used their best endeavours to render the forthcoming meeting worthy of its predecessors in all technical and scientific directions, and numerous technical communications were promised. The local organising committee, composed of Russians belonging to all technical circles, drew up a programme which included excursions to all parts of the country. Suddenly, on February 8th 1904, the very day on which a meeting of Council took place in Vienna, the Russo-Japanese war broke out, seriously interfering with the plans of the Association and rendering doubtful whether the St. Petersburg Congress could be held.

At first, as members of Association are aware, the St. Petersburg Congress was postponed for a year, but afterwards, to the great regret of our Russian colleagues, it was abandoned altogether; in the hope, however, that one of the next Congresses might be held in St. Petersburg. On the present occasion we are happy to meet in Brussels, convinced as we are of the fact that every part of the world into which the idea of carrying out international studies of constructional materials has penetrated, is worthy to become the host of a Testing Congress.

On January 31st (o. s. 18th) 1905, death deprived us of the valuable services of our President, Professor Tetmajer, the most earnest member of committee No 22 which was so closely connected with the proceedings of our earliest Conferences.

Owing to the multifarious nature of the matter under consideration, the Chairman of committee No 22 felt it desirable that the members should not merely express their views in writing, but should be given an opportunity of discussing them by word of mouth. This, however, was difficult to compass, for every nation affiliated to the Association had its representatives on the committee. No other course, therefore, remained open to the Chairman than (1) to cell a meeting of committee No 22 at Vienna on a date when the Council of the Association was sitting (for most members of Council were fortunately also members of the committee), and (2) to utilise the visits he had to pay to Berlin and Paris as occasions for securing personal interviews with the different members who always reside in these cities.

During the period 1901—1905 the author was thus enabled to have two meetings in Paris with MM. Sauvage (Vice-Chair-

man), Le Chatelier, Baclé, Mesnager, and Candlot, and three meetings in Berlin with Herren Martens, Gary, Rudeloff, and Hein. These interviews did much to clear up different points connected with the testing of metals and hydraulic cements, and so greatly assisted the deliberations of the comittee itself when, as just mentioned, a meeting was held in Vienna concurrently with the meeting of Council. The Russian members, MM. Antochine, Baykoff, Bogouslawsky, Charnomsky, Konosiewicz, and Taguéeff Gr, appointed a sub-comittee which afforded the author much assistance, especially at the time when the various recommendations relating to the testing of metals were being finally compared and made ready for press.

Most of the remaining members who were unable to attend any of the mettings referred to laid their views in writing before the Chairman, as is set forth in the report of the committee meeting held in Vienna.

General meetings of the committee took place in 1902 and 1903 during the months of March, at the former of which some very useful suggestions of a general nature were made by the Chairman respecting the future work of the committee and the best method of bringing it to an early end.

At the 1902 meeting it was resolved, on the motion of Professor Martens:

a) To hold the next meeting on the prospective date of the Council meeting, and to study the task imposed on the committee point by point;

b) To ask the members of the committee carefully to read the reports of the two sub-committees (on metals and on cements), together with the proposals made by the German an the American engineers; and to communicate their observations thereupon to the Chairman before the next sitting of the committee.

The Chairman of the committee requested the President of the Association to take the necessary steps for the reconstruction of the committee, and for the translation of the recommendations made by the German and American correspondents.

When the Chairman was in Paris in October 1902, he was enabled, through the courtesy of Professor Le Chatelier, to discuss the existing position of affairs with the French members, viz. MM. Sauvage, Vice-Chairman, Baclé, Candlot, Mesnager, and Le

Chatelier — M. Feret being absent from town. After the matter had been thoroughly discussed, the members present unanimously passed resolutions («Resolutions of the French Members») to the effect that:

a) The proposals emanating from sub-committees «A» and «B» are the result of most exhaustive study on the part of the members, and constitute an extremely valuable piece of work on which all future investigations should be based. Any fresh consideration of the principles underlying the problem before committee No. 22 would only cause an indefinite delay in its solution.

b) In the opinion of the French members, the proposals of sub-committee "B" dealing with hydraulic cements, should be adopted, and only require to be amplified by the addition of regulations (1) respecting plastic cements, such as figure exclusively in the new French rules; and (2) relating to normal sand, if the discussion of this material is brought to a conclusion by the thime of the St. Petersburg Congress.

3. The French members of the committee regard the proposals of sub-committee "A" dealing with metals from different standpoints, certain members having expressed a wish that the proposals should be altered or briefly extended in such manner as to bring them into more perfect harmony with practical conditions (as is the case with the proposals of sub-committee "B"); while others, foreseeing the impossibility of controlling the different qualities of metals that are required in their different applications with the aid of a limited number of tests, suggest that the proposals of sub-committee "A" be accepted in their entirety, and be put into the form which the next deliberations of the committee may show to be most convenient.

The next Meeting of the Committee was held at Vienna March 11th 1903.[1])

When the committee met, the Chairman of the Russian group of members (who were mostly connected with the Mechanical

[1]) All the arrangements for this meeting were made by Herr F. Berger of the Municipal Architect's Office, Vienna, to whose kind assistance his fellow members are greatly indebted. The members present were MM. Belelubsky, Martens, Berger, Bienfait, Hannover, Kirsch, de Mattei, Schüle, and v. Tetmajer; Miss Reinberg of St. Petersburg and Herr Reitler of Vienna acting as secretaries.

Laboratory of the Russian Imperial Institute) redeemed his promise to prepare and to give a digest of all the material that had been received to date, so that the members might be in a better position to judge whether the various suggestions made by different persons could be brought into unison.

I Hyraulic Cements. — Depending upon the proposals of sub-committee "B" the meeting was asked to consider:

a) The already mentioned resolutions of the French members which recognised the great value of the reports presented by both sub-committees, and proposed that they should be taken as the bases for further worth;

b) Some written communications received fromm MM. Gary, Eger, and R. Dyckerhoff.[1]) A lively debate ensued upo the report of sub-committee "B" in which all present took part. The report was finally accepted unanimously, and the following resolutions were also passed:

"That the method of testing plastic cements officially recognised in France be introduced among the proposals of the subcommittee in its proper place, and in the words used by the French members to describe it; and that the Chairman of committee No. 22 be requested to nominate a special committee to study it."

With reference to the definition of normal sand, it should be remarked that the investigation of this question is still in progress. At the suggestion of the President, it was resolved:

"That the necessary paragraphs of the sub-committee's proposals be altered to bring them into agreement with the resolution passed at Budapest respecting the adoption of an international normal sand; and that attention be called to those commissions or referees charged with the study of this matter or an other connected with it. This is the case in problems No. 9 (strength of hydraulic mortars), 29 (weight of cement and standard sand), 30 (finest particles in Portland cement), and 32 (constancy of volume of cement)."

The suggestions made in the letter received from Herr Dyckerhoff upon this subject were examined with particular care.

[1]) In a special letter, M. Feret signified his adhesion to the opinions of his French colleagues.

His suggestion relating to the definition of "Portland cement" was rejected on the ground that former Conferences had already passed resolutions respecting the terminology of cementitious materials. His suggestion referring to paragraph 4 of the proposals was also rejected, because committee No. 22 had already studied the question of normal sand, and had deputed further consideration of the matter to a special reporter. A part of his suggestion about paragraph 6 (binding tests) was rejected because a committee had already been appointed to deal with it; but the other part led to the passing of a resolution:

"That the Council be invited to nominate a committee to consider whether a table should be drawn up for the purpose of reducing the duration of the process of setting to a standard temperature".

A suggestion to pass for international adoption the new German rules covering the mechanical preparation of briquettes containing one part of cement to three of sand used in traction and compression tests, was postponed for committee No. 22 to decide at its next meeting, a printed copy of the rules being handed to each member in the meantime. The suggestion relating to tests of neat cement was rejected, this matter being already part of problem 29.

II. Mechanical Examination of Metals and Alloys. — A preliminary examination of the proposals made by sub-committee „A" in which much assistance has been given to the Chairman by Herr G. Tagueiff, shows that it will be possible to effect a compromise between the said proposals and the different sections and paragraphs of the German Association rules.

Without entering in the present report into a detailed comparison of the paragraphs (see the minutes of the committee meeting for this), it must be sufficient to say that many paragraphs in the two documents are practically identical. This agreement is due to the fact that the French sub-committee (with MM. Hannover, Roussel and Alpherts) had before them the observations made by the German members, which observations served later on to form the basis of the rules drawn up by the German Association.

The paragraphs were divided in 3 sections.

Section I contains 88 paragraphs, sensibly identical in both documents.

Section II contains about 40 paragraphs, with very few real differences. This section requires only a little reconciliation and revision of an editorial character.

Section III contains (a) those paragraphs which are present in the committee's proposals but are partially or wholly absent from the German rules; and (b) those which are present in the German rules but are absent from the committee's proposals.

On the motion of Professor Tetmajer it was therefore resolved that:

»The first section of the paragraphs be passed without further discussion.«

»The second section be submitted to revision by a small sub-committee.«

»The third section be taken into proper consideration at once, in order to settle which paragraphs shall be deleted and which allowed to remain.«

Accordingly, the committee discussed the third section paragraph by paragraph, passing special resolutions on each. The resolutions were then handed to the new sub-committee for editorial revision. This sub-committee was composed of Professor Schüle of Zurich and Professor Kirsch of Vienna. Professor Schüle very kindly also offered to translate the final recommendations into French.

At the wish of Professor von Tetmajer, it was decided to prepare, as an introduction to the report of committee no. 22, an account of its formation and work, in order that the work done by the members might be made public. It was also decided to insert a reference in the report to every problem set by the International Association which deals with connected subjects, and which has been submitted to a committee or to an expert. These problems relate to:

2. Homogeneity of iron;
5. Standard rules for testing;
6. Visual examination of structure;
25. Test methods for cast iron;

26. Notched bars;
27. Ball drop tests;
28. Magnetic and electrical properties;
24. Nomenclature of iron and steel.

By this it should be pointed out that the proposals are confined to resolutions passed by former International Conferences and Congresses.

Professors Schüle and Kirsch have now concluded their laborious task of completing the proposals of sub-committee "A" in accordance with the request made by the committee at its meeting held on March 11th 1903. Hence a new draft scheme for the testing of metals has been prepared in two languages (and more recently also in English), entitled:

Proposals of sub-committee "A", revised by sub-committee "A_2" (Herren Kirsch and Schüle), and brought into agreement with the resolutions passed by committee No. 22 at its sitting of March 11th 1903; together with recommendations relating to the points still remaining unsettled.

These propositions were immediately printed in German and French. In 1906, to the Congress, an English edition was published.

The working out of these proposals greatly facilitated the work of the Commission and it was to be hoped that the proposals of the Sub-Commission would receive the assent of the General-Commission. Messrs Alexandre, Alpherts, Baclé and Hannover, on the one hand, and Messrs Bienfait, Candlot and Gary on the other hand had already succeeded in combining in the proposals A and B the technical opinions of the different countries. Thus the reporting Chairman, at the above-mentioned consultations with the Berlin and Parisian members as well as at the sittings of the General-Commission, had an opportunity of finding out the course which the treating of problem 22 had to take in order to be in harmony with the efforts of the different countries.

The Proceedings of the Commission meeting held in Vienna in March 1903 was sent to all members of the Commission in either German or French. The warmest thanks are due to Herr Geheimrat Martins for editing the former and to Herr Sauvage for the latter.

The proposals of the Sub-Commission, of the same year were treated in the same manner and sent to all members of the Commission together with a circular by the Chairman with a request for individual opinions.

Several valuable answers were received, but as numerous replies were missing, a second circular was issued. The sending out of the same was somewhat delayed owing to the reporter being so much occupied during these last few years, but it may still be possible that the opinions of all the members of the Commission, the American included, will reach the Chairman in time to be discussed at the meeting of the Commission planned for the 2nd September, the eve of the opening of the Congress in Brussels. The answers received from Messrs Baykoff, Antochine, Kirsch, Hannover, Petersen, Charmonsky, Gary, Camerman, Feret, Mattei, R. Dyckerhoff, Bienfait, Schüle, Berger, Dormus, all more or less expressed assent and only a few changes were proposed. These proposed changes are to be discussed at the meeting of the General Commission on the 2nd September in order to come to a unanimous conclusion and to lay their final proposal for a uniform method for testing, before the Congress. Thus one of the most important problems of the Association will be solved which may again form the basis for new problems in the testing of materials.

The author will therefore place the following proposals in the name of the Commission 22 before the Congress.

1. The Congress is willing to accept the proposals A and B (which have been printed and sent to all members of the Association) together with the changes to be finally concluded by the Commission on the 2nd September in Brussels as the solution of problem 22 for metals and hydraulic cements.

2. The Congress expresses its warmest and most grateful acknowledgement for the extraordinary services rendered by the late Chairman H. Polonceau, as well as the former Chairman prof. Debray, and the authors of the initiative works and proposals for the solution of problem 22.

3. The Congress will decide whether the Commission is to be entrusted with the further working out of the proposals presented or whether they may consider their task concluded.

The reporter, as Chairman of the Commission, must in his own name beg the Congress to pass a vote of warmest thanks to the worthy vice-chairmen Messrs Geheimrat Martens and Sauvage as well as to all members of the Commission who have shown themselves co-workers of the Commission in the true sense of the word. And finally with heartfelt feelings let us think of those two men who participated body and soul in furthering our work which to a certain extent, brought countries into closer contact. Two staunch leaders, the never to be forgotten Bauschinger and Tetmayer have accompanied us a long way in our work, and although they are no longer among us may still be considered as having a large share in our efforts.

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INTERNATIONAL ASSOCIATION FOR TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
ANNEX TO REPORT OF COMMITTEE 22.

ANNEX TO THE REPORT OF COMMISSION 22 ON UNIFORM METHODS OF TESTING MATERIALS.

PRESIDENT OF COMMISSION:
N. BELELUBSKY.

VIZE-PRESIDENTS:
A. MARTENS — E. SAUVAGE.

A. MECHANICAL TESTS OF METALS AND ALLOYS.

A) PROPOSAL OF SUB-COMMITTEE A 1 REVISED BY SUB-COMMITTEE A 2 (KIRSCH-SCHÜLE) AND BROUGHT INTO AGREEMENT WITH THE DECISIONS OF COMMITTEE 22 AGREED TO AT THEIR SITTING OF MARCH 11, 1903 TOGETHER WITH PROPOSALS IN REGARD TO THE POINTS WHICH HAVE NOT YET BEEN SETTLED.

B. HYDRAULIC MORTARS.

B) PROPOSALS OF SUB-COMMITTEE B FORMULATED IN THE YEAR 1899 BY MESSRS BIENFAIT, CANDLOT AND GARY, ON THE BASIS OF THE CONCLUSIONS PREVIOUSLY ARRIVED AT.
(TRANSLATED BY R. N. LUCAS.)

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The following problems connected with the problem of Commission 22 are independently treated by commissions or referees:

Problem 2 (Homogenity of iron and steel).
„ 5 (Standard rules for piece tests).
„ 6 (Macroscpic studies).
„ 25 (Test of cast iron).
„ 26 (Notched bars).
„ 27 (Bill pressure tests).
„ 28 (Magnetic and electric properties).
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Proposals for mechanically testing metals and their alloys.

I. General.

1. To the results of the experiments and tests are to be appended complete data in regard to the nature of the material, its source and origin and the method of treatment which is being employed in preparing the test-pieces for experiment; in addition where possible data are to be included regarding the mechanical, physical, microscopic, and chemical qualities of the same.

2. Every apparatus employed for making the tests must be so constructed as to be capable of having its accuracy readily tested with certainty.

15. The machines and instruments must be repeatedly tested for accuracy.

2a. For scientific tests the highest degree of accuracy attainable under the prevailing conditions should be aimed at.

3. The instruments or machinery employed for ascertaining the accuracy of the testing machines must in every instance possess a higher degree of accuracy than the machines themselves.

4. For practical tests the accuracy of the testing appliances and the data furnished in regard to the tests need not be carried beyond the limits of ordinary experimental error due to imperfections in the material.

5. In general an accuracy of from 1 to 2 per cent is sufficient.

7. In giving the results of the tests the fullest data obtainable in regard to the nature of the appliances used and the methods of testing adopted should be given, so as to enable the tests and their value, i. e. degree of accuracy to be estimated.

II. Methods.

A. Strength Tests.

a) General.

8. The materials in the first instance are to be tested to the extent of enabling it to be ascertained whether they are suited to the requirements to which they are in practice to be put.

10. For metals mainly employed for structural purposes the rapidity of the experiment, that is to say the time occupied in performing it, should be moderate, as otherwise disturbing influences may make themselves felt.

b) Tests under steady strain.

1. Machines with their parts.

a) Machines and Measuring Instruments.

12. The testing machines must be so constructed that with careful handling they are „dead-beat".

Arrangements for Fixing.

16. The arrangements for fixing must be such that the tension or pressure is equally distributed over the section of the test-piece.

17. For this purpose it is essential that

a) in pressure-tests free and equable movement of one of the smooth surfaced pressure plates takes place on a ball-socket bearing;

b) in tensile strength-tests free and easy movement of the rods for automatic adjustment at the commencement of the test is essential.[1])

[1]) In this passage only pressure and tension-tests are discussed as the other tests, i. e. bending, notching, twisting, shearing and punching tests are seldom generally used and are only employed for special purposes.

18. For tensile strength-tests experience has shown the following requirements to be necessary

a) In the case of cylindrical rods through ball bearings with an undivided ball bearing.

b) In the case of flat rods an insertion hole and bolts must be employed, when the holes are exactly in the middle line of the rod.[1])

Automatic Recorders (producing lines and diagrams).

21. Automatic recorders must possess the same degree of accuracy as the machines and measuring instruments (see section 5)[2])

2. Test-Pieces

Taking the Test-Piece.

23. In general the test-pieces as far as possible are to be taken from those portions of the material which are put to the severest work in actual practice, or wich conspiciously possede the worst quality.

The selection of the test-pieces must be carried out with special attention to the quality of the materials and must be done later separately for the different materials used.

Form and Structure of the Test-Pieces.

Pressure-Tests.

24. The pressure surfaces of the test-pieces must be as near as possible even and parallel to one another as also at right angles to, and concentric with the axis of the machine.

25. When possible the shape of the test-piece selected for the pressure-test should be in the form of a cube.

26. When the square section cannot be provided it is advisable to make the length l of the prism $= \sqrt{f}$ (f = size of section).

[1]) Ends or heads with milled keyways or slots (to correspond with keys) notched keys or keys with roughened surface when employed are very liable to give rise to unequal distribution of the load but are very convenient in practice and in general sufficient for the purpose required.

[2]) To give a general idea less accurate automatic recording instruments may be employed with good results.

Tensile-Tests.

28, 29 et 30. By straitening material the selection of the length l as compared with the cross section f is of great influence on the elongation. It should be selected in accordance with the formula $l = n\sqrt{f}$ and it is advisable to select the value of n as 11·3 as in many countries a round bar of 20 mm = $^3/_4$ of an English inch and 200 mm = 8 English inches are employed. In all cases the length should be an exact number of centimeters, and n should be given as index to, for example, $\delta_{11\cdot3} = 10\cdot9\%$.

Note. In the case of especially small rods smaller values than 11·3, in accordance with the law of proportionality, should be taken.

For thicker rods than the standard size, lengths of 20 cm may be kept.

32. In flat rods thickness should bear to width the proportion of 3 to 4 on account of the requirements possible to avoid the influence which has square form on the straiteness of material.

34. In the case of test-pieces with heads or expansions the length of the prismatic portion of the rod should be sufficient to exclude the head from exercising any influence upon the test, and the distance between the jaws of the testing machine should exceed the length of the test-piece:

in the case of round rods at each end by one diameter

in the case of flat rods at each end by the breadth.

35. In rods provided with a head or expansion it is advisable that the enlargement to the full size of the head should be a gradual one.

3. Method of carrying out the tests.

Pressure-Tests.

36. No special direction for carrying out the compression-test is needed.

Tensile-Tests.

38. In the case of simple fracture-tests the points to be determined are:

a) Elongation,

b) limit of fracture,

c) elongation up to fracture,

d) the permanent contraction at the fracture,

in a complete test in addition are to be determined,

e) the elastic limit,

f) the limit of proportionality,

g) the coefficient of elongation (or modulus of elasticity).

40. Instruments of precision employed for determining the limit of elongation and proportionality must be such as to indicate $0{\cdot}0005\%$ with accuracy.

41. The limit of elasticity is to be regarded as the degree of tension up to which the material can be considered as still possessing for all practical purposes its full degree of elasticity.

This limit can be assumed as being reached when $0{\cdot}001\%$ of permanent deformation has taken place.

42. The limit of proportionality is to be regarded as the degree of tension at which equal increments of 100 kg/cm² in the tension will produce equal variations in the elongation; each further deformation in this case is to be regarded as equal to those which have previously taken place as long as they do not differ from those which have preceded them by more than $0{\cdot}0005\%$ of the elongation.

43. The permanent elongation is for practical purposes to be measured with an instrument which permits of the alterations in length being estimated to at least 1%.

44. The limit of permanent elongation is formed by the tension at which the increment of elongation remains constant between 0·2 and 0·5.

Note. The limit of permanent elongation is most conveniently determined by means of a measuring rod divided up into percentages of the length to be measured. With many materials the standing still or falling of the indicator of the balance or manometer employed indicates the limit of elasticity with sufficient accuracy.

45. The breaking-strain is the highest tension registered during the test.

46. The tensions on the E. P. S. and B. limits must be indicated within 10 kg/cm².

47. The breaking-strain or strength of the material should be reached by stages in which each increment of tension does not exceed 50 kg/cm².

48. Elongation is to be ascertained either by measuring the lengthening out of the portions of the test-piece from the edges of the fracture to the end marks of the measured portion, or by measuring the distance between divisions previously marked on the prismatic portion of the rod

49 and 50. In the first case those rods are to be excluded from the tests for determining the elongation in which the fracture occurs within a quarter of the whole length of the rod from one end.

51. In the second case owing to the almost exact symmetry of the alteration of form on either side of the fracture the elongation that preceds fracture can always be ascertained as if the fracture ares place in the centre of the bar.

Note. For example in the case of one of the test-pieces having a length of 18 cm, 9 cm of the original, were measured on either side of the fracture. Nine centimeters are to be measured off from the next notch and the length of the cm in which the fracture occurred measured from both sides or parts of the fracture to the next marks.

If the fracture on one side is so near the head that 9 cm cannot be measured and if we suppose between it and the head of the test-piece is only 5 cm., then instead of taking the 4 cm that are symmetrical to the fracture the other 4 cm on the other side should be taken.

52. For measuring the elongation it is sufficient to employ a division on each bar or rod.

c) Impact Tests.

Impact Apparatus.

54. The percussion-tests are to be carried out with the percussion machine which corresponds to the following requirements.

57. The ordinary weight of the „monkey" is 1000 or 500 kg. In special cases smaller monkeys may be employed.

58. The substance of the „monkey" may consist of cast iron or it may be a steel casting or forging.

59. The shape of the monkey is to be so chosen that its centre of gravity lies as low as possible.

60. The gravity axis (line of direction) of the monkey must lie in the middle of the guide.

Note. Percussion machines giving a drop of 6 m can be more easily used in closed premises and can be more reliably built than higher ones; it is advisable therefore in new works not to use machines exceeding 6 m in height and to construct the machine of iron throughout.

61. Special marks arranged on the anvil should indicate exactly on what point the monkey will fall.

62. The proportion of the length of the guides for the monkey to the width between the guides should exceed the ratio 2 : 1.

63. The guide rails should be arranged vertically. The friction should be reduced as far as possible by polishing the surface of the guides where contact is made with them. Lubrication with graphite is recommended.

65. A special hammer track of forged steel should be inserted and secured by dovetailing in a position exactly parallel to the gravity axis of the bear. A special mark should be employed to facilitate the erection of the machine in a correct way as far as this point is concerned.

67. The hammer track should be rounded off to a radius of 150 mm. In the case of percussion-tests in which it is necessary to employ a piece of metal to receive the impact of the monkey and to fix the same by suitably shaping it to the top of the object to be tested as in the case of testing tyres for wheels by the percussion-test, it is essential that the piece of metal that is to be mounted on the tyre is provided at the top with a flat percussion surface.

68. These percussion pieces must be light and where feasible not weigh more than 20 kg.

69. The releasing mechanism for the weigth must be so constructed as not to have any effect upon its free fall.

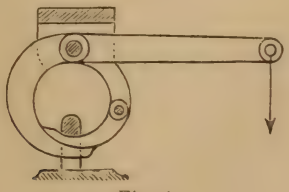


Fig. 1.

71. Arrangements must be adopted which render the accidental falling of the weight impossible.

72. The anvil should be of a piece of cast iron and should be at least ten times as heavy as the weigth.

73. The foundation should be as inelastic as possible and should consist of a compact mass of masonry, the size of which is determined by the general arrangement (of the foundations of the building where it is mounted) that should be at least six times the size of the anvil.

74. The plates bosses or the like employed to support the test-pieces on the anvil are to be firmly secured to the anvil.

75. Arrangements should be made to prevent the test-pieces jumping off their supports but without affecting the impact to which they are subjected.

77. Dividing the heights from which the weigth falls by means of a moveable indicator marked off in centimeters is essential.

2. Method of carrying out the tests.

78. It should be first of all ascertained that the guides and frame in which the monkey moves are perfectly vertical and that the weight has an unobstructed drop.

79. The test-piece is to be so adjusted that the point at which it is struck is exactly in the middle of the space through which the monkey falls.

Note. The height from which the monkey is to be allowed to drop and the weight which it should possess must be specially indicated since the effect of the percussion depends entirely on these two factors.

B. Technological Tests.

a) Bending Test.

90. The bending test must be carried out with a slow and steadily working machine.

92. The machine should bend, viz:

a) either a rod laid upon two supports, the pressure being applied in the middle, or

b) the bar to be tested should be held firm at one end and subjected to lateral pressure.

93. The bending may take place, viz:

a) either freely over a rounded edge (mandril) of the pressure device or the jaws which secure it in position (section 92, *a* and *b*) or

b) held firmly in position and bent round the mandril.

94. In making the bending test with the mandril it is recommended that a mandril of a diameter bearing a proper relationship to the material to be tested should be employed and the thickness of the test-piece may be measured for this purpose.

96. It is recommended that the rods should be four times as broad as they are thick.

97. In the case of test-pieces of section-iron the dimensions of the sections should be maintained as far as possible.

98. The edges are to be rounded off in the middle of the test-piece.

99. For judging the qualities of the material it is best to observe the relationship between the thickness and the semi-diameter of the curve to which the test-piece is bent before noticeable cracks begin to be formed.

100. As a standard of quality the bending coefficient

$$Bg = 50\frac{a}{\rho}$$

should be adopted.

102. The radius of curvature is most easily ascertained on the side towards which the bar has been bent and should be ascertained with scales applied to the side of the test-piece.

103. The radius should increase in steps of ρ of 2 to 2 mm.

104. The opening should always be 45^0.

105. The bending test should be carried out as follows:

a) in the condition to which the goods were supplied to the consumer; and the test-pieces should be cut out cold and so that no alteration of form is caused (not with the shears cf. section 135),

b) after annealing; when the heating has taken place it should be arranged that the iron is heated up to 800^0 C and cools slowly from bright red heat as is done in the case of copper, see section 180; the treatment must in every case be adapted to the material employed

c) After chilling i. e. heating to about 800° C and plunging into water at a temperature between 15 and 30° C and increasing the rapidity of cooling by stirring round rapidly in the water.

106. Cold bending tests are carried out with the material cooled either naturally or artificially to 10 to 30° C. or to zero C.

107. The warm bending test can be carried out either with the metal heated to a blue or red heat; in the first case the iron must be so strongly heated that it becomes coloured blue on any parts of it that have been polished (it does not become blue when it is too hot and does not continue blue when it is too cold); in the second case it must be heated until it appears a dark red in the dark (about 600° C.).

108. The bending test can also be untertaken with test-pieces that have been notched or perforated.

109. The notching must be performed on the side of the test-piece which it is intended shall become convex on bending and the notch should be about 0·2 of the thickness of the test piece and made by means of a rectangularly pointed steel instrument in a planing machine.

110. Perforation is effected when feasible in rods the width of which is five times their thickness and is performed by a stamp of double the thickness of the test-piece.

b) Smith's Tests.

1. Hamering out tests.

111. Drifting-out tests are made either with a hand hammer, a rivetting hammer or a quick acting light steam hammer. The hammer edge should have a diameter of 15 mm and be arranged across the direction in which the test-piece is to be hammered out.

112. The test-piece should be three times as wide as it is thick and hammered out to one and a half or twice its original thickness.

113. The extent to which the test-piece is hammered out from b to b' or l to l' is in the case of where cracks have taken place a value which reaches $\frac{100\,b'}{b}$ or $\frac{100\,l'}{l}$.

2. Setting up tests.

114. »Setting up« tests are carried out with a hammer or quick-working steam hammer.

115. The test-pieces should be as nearly cylindrical as possible, their height should be twice their diameter.

116. The degree of setting up is measured by the extent to which the test-pieces are diminished in length before cracking takes place.

3. Punching Tests.

117. In the perforation-test the test is made on the anvil to see how far from the edge a red-hot test-piece the breadth of which is more than five times its thickness can have a hole punched in it by a punch, the diameter of which is equal to the thickness of the test-piece, without the punched hole breaking away.

118. The resistance to fracture (in this case) is measured by the ratio of the thickness of the test-piece to twice the distance of the hole from the edge.

4. Drifting-out Tests.

119 and 120. In the drifting-out test the test-pieces are heated bright red and their width should be about five times their thickness. A hole is first punched in them with a punch the diameter of which is about twice that of the thickness of the test-piece, and the hole thus formed is drifted out until cracks begin to show themselves.

121. The drifting-out mandrils should increase 1 mm in diameter for every 10 mm of length.

122. When it is necessary to re-heat the test-piece the drifting-out punch should be inserted alternately from either side.

123. As measure of the degree of drifting-out the value $d'/d \times 100$ in which d is the diameter of the hole first punched and d' the diameter to which it is enlarged.

III. Special directions for tests for particular purposes.

A. Malleable Iron.

a) General.

135 and 136. The test-pieces should as a rule be cut out cold with cutting tools avoiding distortion as far as possible.

137 and 138. If the test-pieces are taken from bent portions of metal or if they are cut by the shears they should if it is impossible to heat them be placed under the press and straightened, or straightened by copper hammers; otherwise they are not to be heated to more than cherry red (about 650° C.).

139. If the test-pieces have been sheared or perforated or chiselled 5 mm along their length should be cut off with cutting tools (i. e. milling cutters or the like)

140. Annealing of the test-pieces before the experiment must take place under special precautions and the heating should be to about 800° C. followed by very slow cooling.

141. The surface with which the test-piece leaves the rolls is to be preserved as far as possible.

142. In the case of cut sheets or plates the test-pieces should be obtained from waste pieces or from the top ends of plates, being cut to the proper size longitudinally and crosswise.

In the case of uncut rough plates or sheets the outside edges for 30 mm from the edge must not be employed as test-pieces.

b) Railway Rails.

143. Railway rails are in the first place to be tested in a percussion apparatus by the percussion bending test (see 54, 57 and 81).

144. Owing to the effect of projecting ends these should be made as long as the profile is high.

145. If further explanations in regard to the structure of the material are needed simple tensile-tests should be made.

146. For the tensile-tests it would be advisable to take the test-piece from the outside of the rail.

c) Axles and Tyres.

147—151. Axles and tyres should be tested by the percussion bending test, the former on supports 1·5 m apart. If further information is required regarding the structure or the composition of the material a tensile strength-test should be carried out.

d) Bridge Construction Iron.

1. Puddled Iron.

152 a. Puddled iron for bridge construction should be examined by the tensile and bending tests.

152b. The bending-test should be carried out with the material cold and hot and with unimpaired profile.

153. Bolt and rivet iron should be subjected to the „setting up“ test at a red heat with the mill skin on it.

2. Ingot Metal.

154. For ingot metal the same rules apply as for puddled iron the only difference being that instead of the bending-test at a red heat the tests made are with chilled samples and at a blue heat and also after they have been subjected to notching or the like.*)

e) Boiler Plate.

1. Puddled Iron.

155. Plates should be subjected to the following tests, viz: tensile strength-test, bending-test with unimpaired profile both cold, bluehot and red-hot, as also hammering- and drifting out-tests.

155a. When boiler plate already forged or shaped is to be tested for the qualities it possessed before such treatment, the test-pieces are to be taken from steel that has not been worked and has remained as straight as possible.

155b. If only a bent portion of plate is available it should be obtained by drilling or cutting out with the chisel or circular saw. In all cases the test-pieces are to be treated with all possible care in accordance with sections 137—139.

156. Angle iron is to be subjected to the following tests: the tensile strength-test, the cold and hot bending-tests, the hammeringout test and the drifting-out test.

157. In addition to the common bending test (cold or red hot and with unimpaired profile) with longitudinal test-pieces from the legs (?), the following additional test is recommended i. e. bending section lengths of 100 mm in length until the two ends of the test-pieces are brought together and separated again until cracks appear.

158. Rivet iron is to be subjected to the simple tension-test, the bending-test (with unimpaired profile).

*) The ball pressure-test and the etching-test as well as the percussion test with notched bars are not taken into consideration as the Commitees dealing with these topics have not yet finished their labours.

2. Ingot Metal.

161. The tests in general should be the same as for puddled iron; the bending tests, however after chilling should suffice.

f) Marine Construction Iron.

1. Puddled Iron.

164. Puddled iron for marine construction purposes should be subjected to the following tests: the tensile-strength-test, the bending test (with unimpaired profile) in the cold (section 106) and red hot (section 197) and the smith's hammering out test.

2. Ingot Metal.

165. Ingots metal for marine construction purposes should be subjected to the following tests: the tensile-strength-test, the bending test in the cold (section 106) red hot (section 107) and after chilling (section 105) as also the smith's-tests.

g) Wires.

166. Wires should be subjected to the following tests: the simple tensile-test, the torsion test and the bending to and fro' test.

166b. For the tensile-test the test-piece should be at least 50 times as long as its diameter.

167. The torsion test is to be carried out in a machine with a free length of wire 75 times as long as its diameter. At least the same length in proportion should be used for tensile-test.

168. The machine should be preferably so constructed as not to oppose any force to resulting variations in length of the wire.

169. The bending tests are to be so carried out that the bend takes place over edges the radius of curvature of which is twice the thickness of the test-piece (see section 94).

170. The bending machine should grasp the free end of the wire in such a way that no longitudinal tension is imposed on the test-piece and that the lever arm is, up to the point at which it is grasped, equal to 15 times the thickness.

171. As measure of the quality of the material is taken the number of times which it can be bent. A full bend is one from the straight condition to 90° and back again. These bends take place alternately in opposite directions.

h) Wire Ropes.

172. The main test for wire ropes is the tensile-strength-test.

173. The length tested should be about 30 times its thickness and at least 500 mm long.

174. The parts of the rope which is to be cut should be previously well lapped at both ends for a length of 25 cm[1].

B. Cast Iron.

The test-pieces should be 110 cm in length. The manner of casting (horizontal, vertical or otherwise) should be indicated. The testing is effected by bending with 1 m length of lever arm and medium pressure. Breaking strain tests and compression tests should be made with the two halves of the broken test-piece. The test-pieces required for this purpose should be finished in the lathe.

C. Copper.

177. Copper should be subjected to the following tests:

1. Copper in plates, sheets and rods should be tested with the tension and bending tests in the cold (section 106) or warm (section 107) the „setting up" test being also employed in the case of rods.
2. Copper in the form of copper wire should be tested by the backwards and forwards bending test and the twisting test (see section 166b and f).

[1] The rope is best secured in position in conical boxes by casting in Wood's alloy (of cadmium 4 parts + zinc 4 parts + lead 8 parts + 15 of bismuth). The lengths to be held in the machine should be selected of 10—12 times the thickness. They should be well lapped with wire before cutting off and likewise before being fixed in the boxes and run in with the metal so that the position of the strands remains unaltered. The ends of the wires should also be tinned so that the Wood's metal „takes" better. The recovered metal is not rendered seriously impure by this process. Thin ropes (15 mm and under) may be fixed between clutch-blocks, provided these latter are at least 20 cm long, the strands of the rope being capped and set up with copper wire the end that is held in the machine being lapped with thin wire.

178. The tests can be carried out either with the metal in the form in which it is delivered or in the „soft“ condition.

179. In order to bring copper into the „soft“ condition it is heated to 600°—700° C. then allowed to cool in the air till it is no longer red, and then chilled in water at about 15° C.

180. The test-pieces should only be cut off cold with cutting tools and subsequent straightening is to be avoided as far as possible.

181. The straightening should be carried out very carefully and as far as possible with wooden or copper hammers.

182. If the tests are to be carried out in the „soft“ condition they may be shaped to nearly the final form of the test-pieces and straightened after warming; to bring them into the soft state they must however be heated once more.

183. The preparation of the test-pieces has a great effect in the case of copper; the last shaping must accordingly be accomplished with great care. Notably the tooling between the shoulders of the test-piece should be carried out without interruption and the last cuts should be very thin.

184. The rods or bars are to be smoothed, rubbed down with emery paper and the edges of the flat rods slightly bevelled off.

185. The cold bending test must not be conducted at a temperature below 10° C.

186. In the case of the hot tests the test-pieces are to be heated in a muffle up to cherry red (about 600° C).

187. The rules previously given apply to carrying out these tests (see B. v. Sections 166—171).

D. Other Metals and Alloys.

188. Metals and alloys should be subjected to the following tests: tensile strength, compression and bending tests as also the hot and cold bending test (sections 106—107).

Hydraulic mortars.

Proposals.

In order to arrive at an international agreement it is desirable to discuss the various topics as shortly as possible and to confine the treatment of them to the establishment of a uniform method of proceedure for dealing with the tests and experiments that have most frequently to be performed; the other experiments will be dealt with in later researches.

These proposals accordingly will only deal with the following points:

1. Fineness of meal,
2. Specific gravity,
3. Standard consistency of the cements or mortars,
4. Standard sand,
5. Standard mortars of cement or lime,
6. Setting tests,
7. Tensile-strength-tests,
8. Compression-tests,
9. Cold water-tests for permanency of form.

1. Fineness of meal.

a) The fineness of the meal is determined by the following set of sieves with rectangular meshes.[1]

No of meshes per sq. cm	No of wires per cm	Thickness of wires in mm	Width of meshes in mm
900	30	0·10	0·23
2500	50	0·07	0·13
4900	70	0·05	0·09

[1] The measurements given for the wires and wire sieves with equalled sized meshes are, as experience has shown, difficult to procure in commerce. The dimensions however should be carefully preserved until better sieves can be obtained such as sheets with perforated holes.

The determination of the fineness of the meal should preferably be effected by means of a special machine, as it is difficult to obtain consistent results with hand sieves at different places. A machine for the purpose should be as simply and strongly built as possible and should shake the sieves a definite number of times in a given interval.

b) The substances to be tested should be sieved through two sieves as follows:

Portland cement on sieves with . . . . . { 900 meshes / 4900 „

Other cements and hydraulic lime . . . . . { 900 „ / 2500 „

c) Amounts of 100 grams should be taken for each experiment.

d) The results of the sieve tests are estimated by the amount of residue that remains on the sieves.

2. Specific Gravity.

a) When determining a specific gravity of materials placed loose or shaken into the moulds a cylindrical meter measure of the same height and diameter should be used.[1]

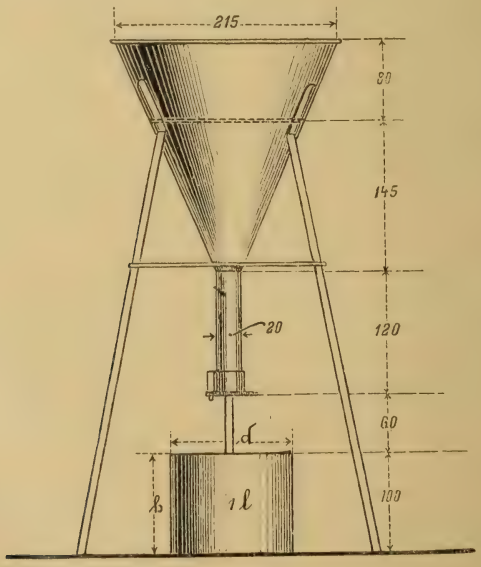


Fig. 2.

[1] A vessel of this sort is not in accordance with the German weights and measures law.

b) Among the apparatus required for these experiments must be included the sieve funnel as illustrated in the accompanying sketch which serves for determining the weight of the amount of powder run in.

In the middle of the funnel is situated a perforated sheet of metal with holes of two mm in diameter. The orifice of the funnel should when full project some 50 mm above the litre measure.

The mortar or binding material is inserted in small quantities of 300 to 400 grams in to the funnel, being finally driven through the sieve by means of a spatula. The filling is stopped as soon as the bottom of the cone which is formed stands level with the top of the measure. Thereupon the surface is smoothened off and the weight of the binding material in the litre measure determined.

c) For determining the weight of the powder when shaken down in the measure an equable percussion movement should be imparted to it. The application of a machine for this purpose is desirable.

3. Standard Grouting.[1]

a) The standard grouting of cement or lime should fulfil the following conditions.

b) The cement or lime, about 40 kg, is put into an enamelled sheet metal measure and the necessary amount of water added to fulfil the following specified requirements.[2]

The grouting is stirred round for five minutes with a broad knife (only one minute when the cement is a quick setting one). The time is counted from the moment at which the water is added.

c) A conical metal vessel with a flat bottom is filled with a portion of the mixed grouting, the dimensions of the metal vessel being 8 cm at the bottom, 9 cm at the top and 4 cm in depth; the top is smoothed off flat with a knife, in which operation sinking of the material and shaking are to be avoided.

d) In the middle of this mass and normal to the surface is placed a dry cylindrical needle of 1 cm diameter and 300 g in weight. The needle is not allowed to fall suddenly, it should be

[1] The term grouting is employed for a mixture of cement or lime with water, no sand being added.

[2] The necessary amount is determined by repeated experiments.

cleaned dry and beof polished metal, its end being cut off at right angles to its axis. The apparatus, called a «Pistill», is so made that it shows the thickness of the grouting between the bottom and the end of the needle.

e) The thickness of the grouting should be such that, at the moment at which the needle stops sinking in, the thickness between the bottom of the vessel and the end of the needle is 6 mm.

4. Standard Sand.

a) The standard mortar should be made with standard sand.

It has been ascertained by experiment that very varying results can be obtained with a quartz sand of a definite size of grain when these sands come from various places, even when their superficial appearance is similar and their impurities are approximately the same. It is desirable that a definite international standard sand should be agreed on. Perforated sheets with round holes are preferable for sieving the normal sand.

5. Standard mortar of cement and lime.

a) The standard mortars should correspond to the following requirements:

For cement mortar and hydraulic lime ought to be employed 1 : 3	250 g cement 750 „ sand
In addition for hydraulic lime mortar 1 : 5 . . .	167 g lime 835 „ sand

Sometimes mixing the mortar with a flat round metal spoon in a smooth dish is to be recommended.

This method of working is recommended in the hope that progress of the experiments may lead to the selection of the better method of mixing. Mixing by machinery is to be recommended.

6. Setting Tests.

a) The setting tests are to be undertaken with the normal grouting as described in section 3. Binding materials, water and air should have a temperature of about 15 to 18° C.

b) The commencement and end of the setting should be determined.

c) As soon as the box is filled it is placed in a case containing damp air at 15—18° C.

d) For the test is employed a metal needle (Vicat's Needle that is cylindrical, smooth, clean, and dry and at the lower end cut off sharp at right angles to its axis. It should be 1 cm^2 in section 1·13 cm in diameter and weigh 300 g.

e) The commencement of setting is regarded as being the moment at which the needle, carefully inserted upon the surface, no longer passes right through the grouting.

The ending of the setting is the moment at which the upper surface of the grouting can support the needle without it sensibly sinking in.

The corresponding periods are reckoned from the moment when the water with which it is stirred comes in contact with the binding material.

7. Tensile-strength-Tests.

a) The tensile-strength-tests for the cem ents should be conducted with pure cement and standard mortar 1 : 3 from the hydraulic limes with standard mortar 1 : 3 and 1 : 5.

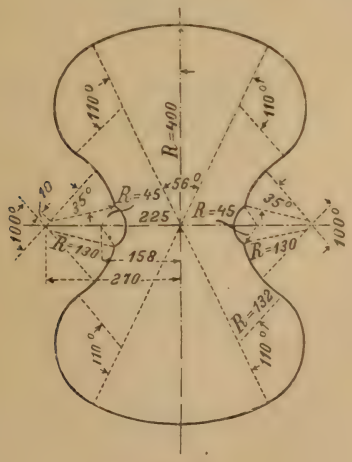


Fig. 3.

b) The test-pieces for the tests should have the shape of the figure 8. These pieces are termed the standard test-pieces, they have a cross-section in the middle of 5 cm². The general appearance is shewn by the accompanying sketch.

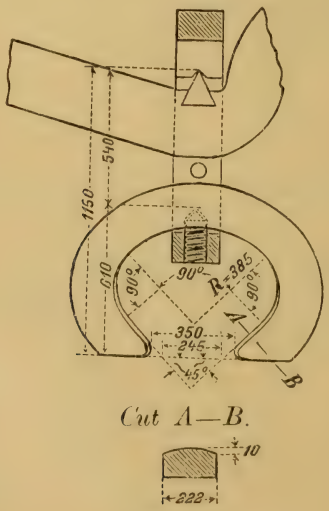
c) The test-pieces 1 : 3 should be finished off in a machine. The machine should be so constructed as to deliver an equal number of blows on each test-piece.

d) The moulds made of iron or bronze for preparing the pure cement test-pieces are laid on a marble table, or on a table of polished metal, after the table has been rubbed over with a greasy cloth.

Into each mould is poured so much grouting as to fill it up and present a slightly convex surface over the top.

Pressure is applied with the fingers so as to ensure that no part is left unfilled, and the mould is struck several times with a mallet on each side in order to let the material settle well in, and to facilitate the escape of air bubbles.

When the cement has contracted somewhat it is scraped off nearly horizontal to the edges of the mould with a straight knife blade so that all the superfluous material is removed without putting any pressure upon it. Finally the surface is smoothed off by means of a knife resting on the edges of the mould.



Cut A—B.

Fig. 4.

e) Another method consists in making a mixture of cement with a small quantity of water till the whole attains the consistency of moist earth; this is pressed into the hole with a spatula Both processes give different results.

f) The test-pieces for the tension-test remain 24 hours after the stirring and mixing on their plates in an atmosphere saturated with moisture, protected from currents of air and direct sunshine, and kept at a temperature of 15 : 18° C.

After this length of time they are taken out of the moulds; the mould is slipped along the plate by unscrewing and removing it from the test-piece. The test-pieces constructed with standard sand must be taken out of the moulds immediately after they are completed.

Mortar made of hydraulic lime must remain for 2, 4 or 7 days in the mould.

g) After these periods have elapsed the test-pieces are dipped in water; the depth of water in the containing vessel should not exceed 1 m.

The water must be renewed each week and kept at a temperature of 15—18° C.

h) The breaking strain apparatus should work in such a way that the increase of load takes place regularly and equally at the rate of 5 kg per second.

The form and method of securing the test-pieces in position by means of grappling claws is shewn in the accompanying illustration.

i) The tensile-tests are carried out in series of 10 pieces after 7 and 20 days etc., counting from the time the materials were mixed.

The mean of the separate values obtained from ten tests is taken as the effective result.

8. Compression-Tests.

a) The pressure-tests are carried out with cube shaped test-pieces each surface of which is 50 cm^2.

b) The test-pieces should be made by machinery.

c) The pressure-test must remain for 24 hours at least in the mould, the mean of 5 results is the figure taken.

d) The tests are to be carried out after the same length of time has elapsed as in the case of tensile-tests (section 7) and should include series of 5 test-pieces (cubes).

9. Testing for permanency of form (Cold Water Test).

a) The cold water tests for permanency of form are conducted with the standard grouting.

b) The grouting is spread out on glass plates. On these it forms cakes, run out thin at the edges, of about 10 : 15 cm in diameter and $1^1/_2$: 2 cm thick.

c) The cakes after hardening for 24 hours in moist air are placed in water of 15 : 18° C.

d) The condition of the cakes is to be observed after the length of time for the fracture and compression-tests has elapsed.

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PRINCIPLES OF A STANDARD METHOD OF TESTING WOOD.

REPORT OF PROFESSOR
□ □ □ MAX RUDELOFF □ □ □
IN GROSS-LICHTERFELDE,

WITH ANNEX I: PROPOSALS REGARDING TESTS FOR ASCERTAINING A PRACTICABLE METHOD APPLICABLE ON A SMALL SCALE TO SHOWING THE RESISTANCE OF WOOD TO PUTREFACTION □ AND ANNEX II: TESTS TO DETER- □ MINE THE DURABILITY OF WOOD, BY
□ □ □ DR. C. VON TUBEUF. □ □ □
(TRANSLATED FROM THE GERMAN-ORIGINAL.)

Principles of a standard method of testing wood.

Report of Professor **Max Rudeloff**, Groß-Lichterfelde.

Translated from the German original.

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P. Welikoff, ingénieur du labor. à l'Ecole Imp. des ing., Moscau.
Ferriev, Professor, Bristol, Engl., University, Rockleaze Avenue.
Filibert Roth, Professor, Michigan, U. S., University.

If the results obtained from the experimenting stations occupied in the testing of wood are to agree generally, it is necessary to have a uniform basis for the method of work.

Therefore, in all investigations as to the technical properties of wood, the following methods are recommended; deviation from them is allowed only if the special object of the investigations requires it.

I. Data on the origin of the materials.

1. To form a judgment of the technical properties of wood the following data on the origin of the test-pieces are required:

a) Designation of the habitat of the trunk, adhering as far as possible to the recommendations of the Association of the German forestal experimenting stations.

b) The quality of habitat, classified thus:
- α) «good» (class I and II)
- β) «medium» (class III) and
- γ) «inferior» or «bad» (class IV and V).

c) Kind of tree, classified thus:
- α) «stood in the open»
- β) «from cleared forest»
- γ) «from uncleared forest».

When selecting test-trunks from an uncleared or approximately uncleared forest the average trunks from 200 of the largest size of the whole forest should be used. If this selection is impossible, the pre-eminent type of trunks only should, be selected for investigation. If this is not done it must be stated, at least, whether the trunks to be tested belong to the largest, medium or inferior class of the forest.

d) The relationships of growth, shown by
- α) the quotient of shape
- β) the shape of the crown (total length of trunk, length of crown and largest diameter of crown),
- γ) the manner of origin,
- δ) the development of the youth and
- ε) the previous economic treatment.

e) The age of the tree (obtained by counting the annual rings of timber on the section).

f) The time of cutting, stating year, month and day.

g) The kind of storage and seasoning undergone between cutting and testing.

h) The position of test-piece in the trunk.

II. Exterior signs of the properties.

2. In the case of each test-piece the following particulars should be stated:

a) According to the appearance of the longitudinal section or the split-surface:

α) the direction of the wood fibres, whether grown straight or crooked,

β) number, kind and position of any existing knot-stumps;

b) according to the appearance of the section:

α) the average radial width of the annual rings,

β) the width of 30 to 30 annual rings, as a measure for equal periods of age, beginning with the outside rings,

γ) the lengths of the annual rings found on 1 cm^2 sectional-surface, determined by a measuring wheel.

δ) the formation of the annual rings, «circular» or «eccentric»,

ε) with conifers, the average proportion of width between autumnal-wood and spring-wood layers. When testing on a larger scale, this condition may be eliminated as the volumetric weight gives already some approximate information on this point.

III. The test-methods.

3. To test the wood thoroughly, the following properties must be ascertained.

The specimens should be free from knots if possible, and in any case the results on knotty wood and on wood free from knots should be kept separate.

A. Strength.

4. The strength of timber is first fixed by a pressure-test, followed by bending- and shearing-strain-tests.

5. The difference in strength, when testing the different parts of trunk-section, the influence of habitat and the conditions of growth are in the first instance to be ascertained by pressure-tests.

6. As the rate at which the load is applied influences the test result remarkably, the load should always be increased with the same speed, if possible by 20 kg/cm^2 per minute.

7. The permanent deformation is only to be recorded if, after one minute's freedom from load, no further change is visible.

8. The moisture in the specimens is to be determined when testing (see paragraph B).

9. If a large number of pieces, similar as regards species of wood, seasoning, and size are tested at the same time, it is sufficient to fix the average moisture of some of these pieces.

10. The figures obtained to show the strength are only comparable if the percentage of moisture in the wood is the same in all cases. It is, therefore, advisable to undertake tests generally with seasoned samples only, to assume 12 per cent as a normal percentage, and, as far as the conditions of the influence of the moisture have already been found, to reduce the figures found to the standard 12 per cent of moisture.

11. On account of the great differences in quality in different parts of the same piece, at least three parallel tests are necessary to obtain average figures.

1. The Pressure-Test.

12. To complete a thorough investigation it is necessary to determine

a) the tension at the limit of proportionality;

b) the modulus of elasticity E, or the coefficient of elongation α; in other words, the shortening of the length-unit for the tension-unit;

c) the breaking-strain (highest tension attainable);

d) the shortening with progressive load up to fracture;

e) the quotient $\frac{\text{compression-strength}}{\text{volumetric weight}}$ with normal moisture.

13. The breaking strain to be determined on cubes.

14. The elasticity to be determined on prisms of square section, their length being equal to the triple sectional edge. They are to be cut as per fig. 2, and then the diminution in the height to be determined separately on the flat surfaces and the vaulted surfaces. The measuring-length shall be equal to the sectional edge and situated symmetrically to the centre.

15. The load is to be applied between flat plates, one of which is movable to permit of automatic adjustment, and should exert its influence parallel to the direction of the fibres (trunk axis) of the wood. Specimens with crooked fibres are to be excluded from the test.

16. The lateral faces are to be planed smooth, the surfaces to which the pressure is to be applied being prepared with especial care. Well guided saw-cuts are as valuable as careful planing.

17. When testing entire trunks, specimens should be taken as mentioned hereafter:

a) to find the average strength as per fig. 1 so that a diagonal of the square pressure-surface forms a radius of the trunk-section;

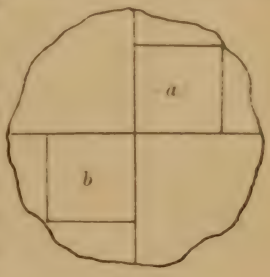

Fig. 1.

b) in special tests for finding the influence of the age of the timber on the strength, as per fig. 2, the test-pieces should be selected so that the two faces are as much tangential to the annual rings as possible.

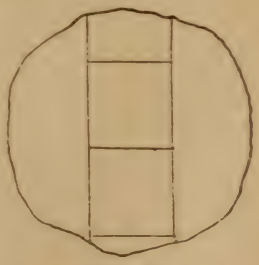
Fig. 2.

18. When taking samples as per *a* at least two test-pieces *a* and *b* (fig. 1) should be selected from each trunk-section, i. e. at the same height and diametrically opposite to one another.

19. If the machine is not strong enough to crush such large squares, they will have to be divided into 4 or, if necessary, 9 equal parts of square section, and from these test-pieces the systematically selected area is to be tested.

20. When taking samples as per *b*, the samples are to be selected from both sap-wood and heart-word, provided the nature of the wood admits a separation with accuracy.

21. The test-samples from individual wood-pieces (cut-wood) are to be selected, as far as the special use and test admit, in such a way that there is no pulp inside the test-piece.

22. The position of the annual rings on the sides, and whether the samples are of heart-wood or sap-wood, is to be stated.

23. If the samples consist of both kinds of wood, the division is to be explained by a sketch (see fig. 3), or the share of both to be estimated.

Fig. 3.

2. The bending-test.

24. For bending-tests prismatic staves or rods are used.

25. The test takes place with both ends supported and a single load in the middle.

26. The local pressure under the load-centre is to be rendered harmless, as far as possible, by applying a rider of hard wood of the following measurements: width = width of stave; length = $^1/_{10}{}^{th}$ of supporting distance; depth = $^1/_3{}^{rd}$ of the height of stave.

27. The ends of staves should rest on rollers, using plane interpositions, the bearing surface of same being equal to the section of stave.

28. When testing entire trunks the bending-trials should be so made that a diagonal of the square-section forms a radius of the trunk.

29. Of 4 pieces cut from the same trunk, 2 at least, situated diametrically opposite, (better all 4) have to be tested.

30. The load is to be so applied that one of the two surfaces, situated radially in the trunk, lies on the tensile side, or the side on which outward distortion takes place.

31. The sectional measurements of the test-pieces depend on the section of the trunk. The distance between the supports should be at least 8 times greater than the height, but generally it is 1·5 meters.

32. From individual pieces of wood the bending-samples are, if possible, to be so selected that the annual rings run perpendicular to one side. When testing, this side is to be considered as the tensile-side.

33. With all bending-tests the relationship of the annual rings to the direction in which the distorting stress is exerted is to be shown, and explained by a sketch, if necessary (see paragraphs 22 and 28).

34. For a complete bending-test the following data should be ascertained:

a) the tension at the proportional limit;

b) the modulus of elasticity;

c) the breaking-strain under greatest load;

d) the course of the deflection with progressive load, up to the greatest load (breaking-strain) for getting the bending-diagram;

e) the work expended in deflexion up to P-line.

f) the work expended in deflexion up to greatest load (break), to be ascertained by diagram.

35. The deflection is to be ascertained within 0·01 mm.

36. The proportional line is the load up to which the compensating line for the diagram runs straight.

37. The strains are to be calculated in accordance with the formula of flexion, provided this formula is valid up to the break.

38. By breaking-load is meant the highest load carried by the test-stave; any breaks of the fibre-bunches or splitting of the surface are not considered as breakages.

39. The work of flexion is to be reckoned for a standard test-stave of 10 × 10 cm section and 1·5 m supporting distance, to make the figures agree for staves of different measurements.

3. The shearing-test.

40. The shearing-test on prismatic test-pieces is to be carried out in one cut.

41. It should be used in ascertaining the strength radially and tangentially to the annual rings.

42 The point of application of the load is always to be from the direction of the face of the section of the trunk.

43. The shearing-jaws shall not be wider than 10 mm, and their acting surfaces should be vertical to the direction in which the force is exerted.

44. The breadth of the samples (shearing-surface), when cut radially, should not be above 5 cm, with tangential cut not above 30 mm.

45. The length of samples in the direction of the force exerted should be equal to four times the breadth of shearing-jaws.

46. The value of the breaking-strain is to be given as for the entire section of the sample; the deformation under the shearing-jaws is to be neglected.

4. The tension test.

47. To find the tensive strength in the direction of the fibres, flat staves or rods are used. They are best made of pieces split so that the broad sides stand either normally or tangentially to the annual rings.

48. The thickness of the staves shall be 1 cm, the breadth at least 2 cm, and the testing-length 22 cm.

5. Split tests.

49. The samples are to have the shape introduced by Nordlinger, as per fig. 4, with split surfaces in radial and tangential position to the annual rings.

50. If te test material does not suffice for the length l Rudeloff proposes iron prolongations (fig. 5).

51. The measurements shall be:

$a = 2{\cdot}5$ cm; $b = 5{\cdot}0$ cm; $h = 5{\cdot}0$ cm and $w = 1{\cdot}5$ cm.

52. The total breaking-load is to be observed.

B. The percentage of moisture.

53. The proportion of moisture is to be indicated as a percentage of the entire dry-weight.

54. The proportion of moisture is to be ascertained immediately after the whole of the samples have been tested. If these are too large for the purpose then the proportion of moisture is to be determined from discs, cut off, if possible, close to the break, and covering the whole circumference of the trunk.

55. These discs to be 2 to 5 cm thick.

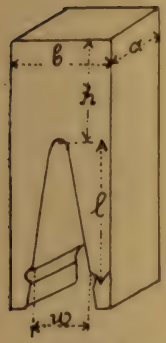

Fig. 4.

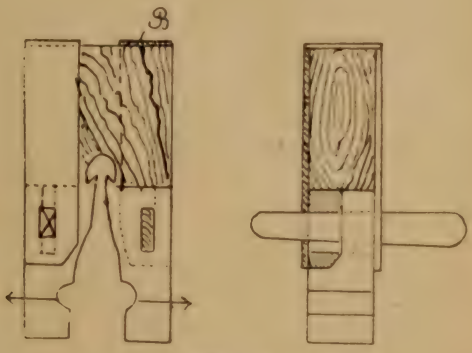

Fig. 5.

56. The discs to be cut off by a hand-saw immediately after the test for strength.

57. The drying is continued, in a well ventilated drying-cabinet having double walls, until there are no greater differences in weight than 0·3% as compared with the dry-weight.

58. The dried samples are weighed only after being cooled down to air temperature in well closed exsiccators over sulphuric acid.

59. The normal proportion of moisture (standard moisture) to which the strength- and weight-figures are to be reduced, should be 12%.*)

*) As regards the normal percentage of moisture the views of the members of the Commission differ. The majority was for 12%, the rest for 15%. This question is therefore to be decided upon at the meeting of the Association.

C. The specific gravity.

60. The specific gravity is fixed either:

a) stereometrically by calculation from measurements of nicely finished samples free of clefts, or

b) by the process of immersion, according to the quantity of water displaced.

61. For the process of immersion the Friedrich precision-xylometer is used.

62. Samples of smaller size, especially thin disc-shaped samples, must be protected against water. This is easily done by a coat of linseed-oil, or by immersion in a solution of paraffin in benzole.

63. In this case the change of volume is so slight that it may be neglected.

64. Larger samples do not require a protection against water, if the immersion be carried out quickly (in about 15—20 seconds).

65. The figures for the specific gravity are to be calculated out on the assumption that proportion of moisture present is uniformly 15%.

D. The shrinking and swelling.

66. The change of the volumetric contents by shrinking and swelling is ascertained either

a) at once by means of immersion of pieces of suitable shape, or

b) by calculating the change of lengths of prismatic samples vertically and tangentially to the annual rings and also in the direction of the length of the trunk.

Besides the results of measurements of shrinking and swelling, the corresponding changes of weight are to be stated.

67. In the case of the methods mentioned under *b* two measurements are to be made in the middle line of each of the six prism surfaces.

The following measurements must be taken, either

a) the total lengths on the middle line of carefully worked prisms, or

b) special lengths which are marked by strokes along the middle line.

68. The accuracy of all measurements must be within 0·1 mm.

69. The breadth of any crevices to be deducted from the legths measured.

E. Durability of the wood.

Definite proposals for uniform tests of the durability of wood cannot be made at present as there are no fundamental experiments on which to base them. Professor Dr. von Tubeuf has undertaken to carry out the preliminary work required in this connection. His reports up to date are in the form of appendices I and II.

IV. The selection of test-pieces from different heights of the trunk.

70. When dividing the trunks, in order to take samples from different heights, care should be taken that the remaining pieces are economically used.

71. The selection of the height, from which the samples are taken, should be in conformity with the purpose of the investigation.

72. To find the average properties of the trunks, the samples are to be selected as follows: one portion should be cut out for the preparation of the test-pieces for the bending-trial between 7 and 10 m, from the ground; the samples for the other tests being selected directly above and below the samples for the bending-test.

73. To test the suitability of the trunks for beams and struts of definite length, the middle of the bending-samples shall coincide, if possible, with the middle of the portions to be employed in practical work.

The other samples are again to be taken from the ends of the testing-pieces.

74. When investigating the influence on the test-pieces of their relative height in the trunk, the lowest portion cut out for ascertaining the compressive strength and the specific gravity shall be 1·3 m above the ground (breast height). The next cuts are then made at 1, 5, 11, 17 m and so on, progressing each time 6 m above the stump-cut, until the diameter of trunk is less than 13 cm.

75. All samples and individual test-pieces are to be designated by the height in the trunk from which they are cut.

76. To determine the average condition of the wood from a distinct habitat, 3 trunks at least should be tested.

Annex 1.

Proposals regarding tests for ascertaining a practicable method, applicable on a small scale, to show the resistance of wood to putrefaction.

By Professor C. von Tubeuf.

To detect the difference in durability of different timber, and of timber impregnated in various ways, no special methods have hitherto been used. If it was a question of sleepers, the timber, suitably impregnated, was merely used in form of sleepers on certain railway lines, and it was observed for how long they continued serviceable. If it was the question of vineyardstakes, the stakes, suitably treated, were used in the vineyard in the same way as ordinary ones and their durability practically tested.

The same course was followed as regards the wood used for timbering in mines.

In all these instances the timber is relatively quickly destroyed, because of its being exposed to the air, moisture and heat.

With stakes the decomposition takes place most quickly at the point of contact with the wet soil. For this reason tests were generally made with stakes put into the earth to find out the durability of the timber. But years pass with all such tests before a positive judgment can be pronounced; and then it is only a practical one, and refers merely to the possibility of using timber for a distinct practical purpose.

Instead of these practical tests another method was sought. Sundry investigations showed that, using the same kind of timber the higher the specific gravity of the wood the more solid and the more durable it was. Therefore the specific gravity was made use of as a direct gauge of the durability of specimens of timber

of the same kind. But in the case of different kinds of wood, or of specially treated (e. g. impregnated) wood, the specific gravity could then no longer be regarded as a gauge; only the mechanical and technical properties of the pieces could be determined, and not their durability under the attack of organisms.

To do this it was necessary to undertake the practical tests mentioned.

To discover how far the durability and the specific gravity of timber are proportionate, it would be advisable to find by special experiment the time of decomposition, particularly of pieces of timber of the same kind but of various specific gravities.

The same experiments should be carried out with timber of different kinds but of particular specific weight.

Experiments already made on the durability of timber showed that the decomposition of the timber was not due to the influence of bacteria but to the activity of higher fungi.

In nature, individual kinds of wood are frequently destroyed by specific kinds of fungi. At the same time there are also fungi which will attack any kind of timber. The most important, the most widely-spread and best known amongst them is the house fungus (Merulius lacrymans). Experiments can easily be made with it as it is easy of cultivation.

We have here a uniform means for effecting decomposition, and it can be ascertained which of the kinds of wood under examination are earliest and latest attacked by the house fungus.

It must be borne in mind that, in nature, there are several fungi acting together, each taking a share in the decomposition of the wood; but as yet we are insufficiently acquainted with their action, and moreover, they attack the wood in a similar manner. Hence, it may be assumed that wood, which is readily attacked by house fungus, would be liable to lose its strength when exposed under natural conditions to the attacks of other fungi.

Although house fungus is not exactly similar to the timber-destroying fungi existing in nature, still it seems advisable to conduct, first of all, experiments with this distinct and widely-spread fungus.

The tests undertaken by me were made in the following manner:

From round timber of different kinds of wood (especially fir), and from places free of knots, 2 neighbouring horizontally cut discs

of nearly the same thickness were sawn off and brought to exactly the same weight by planing the cross-surface, or by working it with a chisel. The volume of both discs was determined, then the dry weight of one of them, and, as a result, the moisture of both discs at the time of the first common weighing. By so doing the dry-weight of the corresponding disc was found without its having been dried.

Having been marked by a certain number of needles thrust into it, it was exposed to fungus-attack. This was done as follows: The house fungus (resp. Polyporus vaporarius) was cultivated in wooden cases, on sawdust and little boards, or on blotting paper, by placing in these receptacles pieces of wood already attacked. It is desirable to have large quantities of growing fungus at hand, and to have the fungus spread out on a plane surface in order to allow the discs under experiment to be quickly and regularly attacked.

After a certain period the exposed disc was taken out. Its moisture and dry-weight could now be determined.

With the aid of the dry-weight determined on the other disc, and the dry-weight now found of the exposed disc, the loss in the weight caused by the decomposition was determined. That this is considerable when timber is destroyed by house fungus, was proved by the detailed and searching investigations made by R. Hartig (Der echte Hausschwamm, Berlin. Published by J. Springer 1885).

It would be advisable, when continuing the tests, to determine not only the specific gravity but also the technical properties (e. g. the crushing-strain) of the discs. In the case of heartwood the splint may, of course, be removed from the discs, the best sort of wood being examined by itself. Parts of the discs belonging to different periods of growth may also be examined separately. Reference must be made to the difficulty experienced in obtaining a rapid and uniform fungus-attack. The experiments could not be completed, as the fungus must be cultivated in a cellar of uniform temperature at all times of the year. In the cellar used by me, the cultures of last summer had entirely ceased growing during the winter.

Annex II.

Tests
to determine the durability of wood.

By Dr. C. von Tubeuf.

In accordance with former statements, it was first determined whether wooden discs of different kinds of wood would in a short time be so decomposed by the fungus that their weights would decrease materially. To ascertain this it was necessary to find the weight after drying of both the sound- and decomposed wooden discs. As it appeared to be of no use to expose the dry discs to the fungus, equal weights of two neighbouring discs, taken from the same branch, were employed. One disc was weighed without fungus-attack, the other after such attack and after being thoroughly dried.

The results are shown in the following table.

Disc No.	Original-weight g	Original-volume cm³	Dry-weight g	Moisture	After Fungus attack			Kind of tree
					Dry-weight	Loss of substance absolute	Loss % of dry substance of the sound disc	
3	72·20	12	52·67	17·53	44·45	8·22	15·61	Fir
4	70·00	11·2	49·79	20·21	45·42	4·37	8·78	„
7	69·2	11·1	47·90	21·30	37·09	10·81	22·57	„
10	89·05	12·8	56·10	32·95	37·93	18·17	32·39	„
	123·7	148·5	73·22	50·48	50·39	22·83	31·18	Birch
	95·2	121·5	51·24	43·96	33·02	18·22	35·56	Alder

The pieces were exposed to fungus-attack from January to October. In winter the cellar, in which the discs were kept, was cold, and the growth of the fungus unnoticeable. The figures prove that the fungus-attack and loss of weight are coincident. It remains to determine whether an ample and uniform vegetation of fungus is obtainable such that discs of the same weight and of the same specific weight show the same loss of substance in the same period. Such tests are now being made with better arrangements.

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 24.

ON THE UNIFORM NOMENCLATURE OF IRON AND STEEL.

REPORT
OF COMMITTEE 24, PRESENTED BY
HENRY M. HOWE, CHAIRMAN,
NEW-YORK, AND
ALBERT SAUVEUR, SECRETARY,
CAMBRIDGE, MASSACHUSETTS.

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On the uniform nomenclature of iron and steel.

Report of Committee 24, presented by
Henry M. Howe, Chairman, New-York, and **Albert Sauveur**,
Secretary, Cambridge, Massachusetts.

(Resolution of Council, February 3rd 1901.)

The Committee appointed for the solution of problem 24 consisted of the following members.

Chairman:

Howe, H. M., Professor, Columbia University; New-York.

Vice-Chairman:

Lévy, L., Engineer; Paris, France.
Tschernoff, D., Professor; St. Petersburg, Russia.

Secretary:

Sauveur, Albert, Professor, Howard University, Cambridge; Massachusetts.

Members:

Van Drunen, Engineer; Ixelles, Belgium.
Tusen, H., Lieutenant colonel; Kopenhagen, Denmark.
Martin, E. P., The Hill; Abergavenny, England.
Pourcel, A., Engineer; Paris, France.

Brauer, H., General manager; Dortmund, Germany.
Wedding, H., Dr. Professor; Berlin, Germany.
Verole, P., Engineer; Milan, Italy.
Baalsrud, A., Engineer; Christiania, Norway.
Dormus, A. R. v., Engineer; Vienna, Austria.
Sailler, A., Engineer; Vienna, Austria.
Bureau des Forges; St. Petersburg.
Belelubsky, N., Professor; St. Petersburg.
Jossa, N., Director; St. Petersburg, Russia.
Korobkoff, M., Major general; St. Petersburg, Russia.
Smirnoff, S., Director; St. Petersburg, Russia.
Tetmajer, Ladislaus v., (†) Director; Budapest.
Campbell, H. H., Supt. and Gen. Manager; Steelton, Pa.

Early in February 1905, a list of species and varieties of iron and steel, with definitions, drawn by the Chairman and Secretary of Committee 24, was sent to every member of this Committee with a request for comments and suggestions. A special request was sent to the following gentlemen for the equivalents in their several languages of these species and varieties.

A. Pourcel, Engineer, Paris, France.
Prof. H. Wedding, Berlin, Germany.
J. A. Brinell, Engineer, Stockholm, Sweden.
Prof. H. I. Hannover, Kopenhagen, Denmark.
Prof. P. Kley. Delft, Holland.
P. Verole, Engineer, Milan, Italy.
Col. Cubillo, Trubia, Spain.

This present report, which is based upon our original list and definitions, and upon the replies to these requests, consists of four parts.

1. A polyglot, which gives the names of the chief classes of iron and steel in English, French, German, Swedisch, Danish and Dutch. We regret that we have failed to get the Italian and Spanish names.

2. English definitions of the principal classes of iron and steel.

3. A glossary of the special sizes and shapes of iron and steel.

4. A note on the boundary between steel and cast iron.

I. Polyglot, in English, French, German, Swedish, Danish and Dutch, of the chief classes of iron and steel, and of the apparatus in which they are made.

This list is not intended to be complete, but rather to include only those varieties which are of interest to the Association.

II. Definitions.

Alloy Cast Irons, those which owe their properties chiefly to the presence of an element (or elements) other than carbon.

Alloy Steels, those which owe their properties chiefly to the presence of an element (or elements) other than carbon.

Basic Pig Iron. In America, pig iron containing so little silicon and sulphur that it is suited for easy conversion into steel by the basic open-hearth process. It is restricted to pig iron containing not more than 1·00 per cent of silicon.

Bessemer Pig Iron, that which contains so little phosphorus and sulphur that it can be used by itself for conversion into steel by the original or acid Bessemer process. In America this term is restricted to pig iron containing not more than 0·10 per cent of phosphorus.

Bessemer Steel, steel made by the Bessemer process, whether its carbon content is high, low or intermediate.

Blister Steel, steel made by carburizing wrought iron by heating it in contact with carbonaceous matter. It might also be made by so carburizing a low-carbon steel.

Cast Iron. Generically, iron containing so much carbon or its equivalent that it is not malleable at any temperature. Specifically, cast iron in the form of castings other than pigs, or remelted cast iron suitable for casting into such castings, as distinguished from pig iron, *i. e.*, cast iron in pigs, etc. (See Pig Iron.) For instance, cast iron pigs, *i. e.*, pig iron, like lead in pigs, *i. e.*, pig lead, is remelted and cast into castings, such as columns, locks, gears, etc., of special shape suited to their special purpose; these are specifically called "cast iron," and this is the usual restricted meaning of "cast iron" in trade language.

When we say "the fracture and density are those of cast iron," "the right-hand side of Roberts-Austen's diagram represents cast iron," or "Production of Steel by the Partial Decarburization of Cast Iron" (Percy, Iron and Steel, XV), we use "cast iron" in its generic sense, including both pig iron in pigs and cast iron in other and shaped castings, whether these are made direct from the molten pig iron running from the blast furnace, or by remelting that cast iron. On the other hand, when we say "cast iron locks, posts, and other hardware," we use "cast iron" in its specific sense of "shaped castings of cast iron," and we exclude pig iron. Exactly in the same way when we say "all men must die" or "the cavalry lacks horses, we use "man" and "horse" generically to include women and mares; when we say "the men are at the war" or "the horses beget colts," we use "man" and "horse" specifically, and exclude women and mares. The meaning of "pig iron," "woman" and "mare" is never in doubt; in which of its two meanings a word like "cast iron," "man" or "horse" is to be taken should be and generally is indicated clearly by the context. Dual meanings might conceivably lead to confusion, but in fact they do not; probably no reader of these present lines has ever been confused, unless intentionally, as to the meaning of "horse" or "man," nor need he be as to the meaning of "cast iron." Were your Committee inventing language, it would avoid dual meanings; as our function is not to invent but to record existing language, we have no jurisdiction over the meanings of these words. We only assert our knowledge that the generic sense of "cast iron" is so firmly established in English literature that it cannot and should not be changed. That the trade itself rarely has occasion to make use of the generic meaning of cast iron would not warrant a vain attempt to root that established meaning out of scientific and literary usage. The trade need never use the generic meaning of "cast iron." The truth is that science and letters often need generic terms which trade does not need; "mollusc" is needless to the fish trade, "mammal" is needless to the circus trade, "conifer" is needless to the lumber trade; nevertheless those words and that sense will remain, because they are needed elsewhere.

The Committee recommends drawing the line between cast iron and steel at 2·20 per cent carbon for the reason that this

appears from the results of Carpenter and Keeling to be the critical percentage of carbon corresponding to the point "a" in the diagrams of Roberts-Austen and Roozeboom. As to the signification of this critical point the Committee is not prepared to express an opinion.[1]

Cast Steel, the same as crucible steel. Obsolescent, and to be avoided because confusing and because a temptation to fraud.

Cemented Steel, the same as blister steel.

Charcoal Hearth Cast Iron, cast iron which has had its silicon and usually its phosphorus removed in the charcoal hearth, but still contains so much carbon as to be distinctly cast iron.

Converted Steel, the same as blister steel.

Crucible Steel, steel made by the crucible process, whether its carbon content is high, low or intermediate.

Gray Pig Iron and Gray Cast Iron, pig iron and cast iron in the fracture of which the iron itself is nearly or quite concealed by graphite, so that the fracture has the gray colour of graphite.

Hematite Pig Iron, originally pig iron made from the hematite ores of England, which happen to be so free from phosphorus and sulphur that the pig iron made from them can be used by itself for the acid Bessemer process. By association it has come to mean any pig iron thus relatively free from phosphorus and sulphur. The term is not used in America, and is undesirable.

Hot Metal or Direct Metal, the molten cast iron from the blast furnace before it has been allowed to solidify.

Ingot Iron[2], steel cast into an initially malleable mass and containing so little carbon or its equivalent that it does not harden greatly on sudden cooling. The word is rarely used in English

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[1] Professor Wedding reports that in Germany every metallic product of the blast furnace is called pig iron or cast iron, and appears to dissent from drawing any line between cast iron and steel. Mr. Brinell thinks that 2·20 per cent of carbon is a rather high limit for practical purposes.

[2] This expression is not used in Denmark where the nomenclature proposed in Philadelphia (1876) has never been accepted (Hannover).

"mild steel" or "low carbon steel" or "soft steel" being generally used in its place. In America the line between soft steel and half-hard steel is usually drawn at a carbon content of about 0·20 per cent.

Ingot steel[1], steel cast into an initially malleable mass and containing so much carbon or its equivalent that it hardens greatly on sudden cooling. The word is rarely used in English, but "hard steel," "high-carbon steel" or "half-hard steel" are used in its place.

Malleable Castings, castings of malleable cast iron, which see.

Malleable Cast Iron, iron which when first made is cast in the condition of cast iron, and is made malleable by subsequent treatment without fusion.

Although the English name of this variety suggests that it is cast iron, it is not truly a variety of cast iron, but rather forms an independent species of iron, because it lacks the essential property of cast iron, *viz.*, its extreme brittleness. Though the term "malleable castings" is very common, the term "malleable cast iron" is very rarely used. The common but inexcusable term we regret to say is "malleable," pronounced "mallable," used as a substantive. Those with some respect for their mother tongue, if asked of what material a malleable casting was composed, would generally use a circumlocution.

Malleable Iron, the same as wrought iron. Used in Great Britain, but not in the United States, except carelessly as meaning "malleable cast iron" (vulgar "malleable").

Malleable Pig Iron, an American trade name for the pig iron suitable for converting into malleable castings through the process of melting, treating when molten, casting in a brittle state, and then making malleable without remelting. The term should be used with care to avoid confusion. This material is also called in trade in America "malleable iron", but this use should be avoided, because „malleable iron" has the older and (in Great Britain) firmly established meaning of "wrought iron".

[1] This expression is not used in Denmark (Hannover).

Mottled Pig Iron and Mottled Cast Iron, pig iron and cast iron the structure of which is mottled, with white parts in which no graphite is seen, and gray parts in which graphite is seen.

Open Hearth Steel, steel made by the open hearth process, whether its carbon content is high, low or intermediate.

Pig Iron, cast iron which has been cast into pigs direct from the blast furnace. This name is also applied to molten cast iron which is about to be so cast into pigs or is in a condition in which it could readily be cast into pigs[1].

Plate Iron, a name applied in Great Britain to refined cast iron.

Puddled Iron, wrought iron made by the puddling process.

Puddled Steel, steel made by the puddling process, and necessarily slag-bearing. (See Weld Steel.)

Refined Cast Iron, cast iron which has had most of its silicon removed in the refinery furnace, but still contains so much carbon as to be distinctly cast iron.

Shear Steel, steel, usually in the form of bars, made from blister steel by shearing it into short lengths, piling, and welding these by rolling or hammering them at a welding heat. If this process of shearing, piling etc., is repeated, the product is called "double shear steel".

Steel, iron which is malleable at least in some one range of temperature, and in addition is either (a) cast into an initially malleable mass; or (b) is capable of hardening greatly by sudden cooling; or (c) is both so cast and so capable of hardening. Variety A includes also molten iron which if cast would be malleable, as do its two sub-varieties, "ingot-iron" and "ingot steel". (Tungsten steel is malleable only when red-hot.)

Steel Cast (adjective), consisting of solid Bessemer, open hearth, crucible or other slagless steel, and neither forged nor rolled: applied to steel castings. For instance, a "steel cast" gun

[1] Molten cast iron is not in Denmark called pig iron (Pigjaern). Hannover.

is a gun which is a steel casting, *i. e.*, which has been neither forged nor rolled. To call it a "cast steel" gun would imply that it was made of crucible steel, to which the term "cast steel" is restricted.

Steel Castings, unforged and unrolled castings made of Bessemer, open hearth, crucible or any other steel. Ingots and pigs are in a sense castings; the term "steel castings" is used in a more restricted sense, excluding ingots and pigs and including only specially shaped castings, such as are generally used without forging or rolling. They may, however, later be forged, *e. g.*, under the drop press, when they cease to be "castings" and become "drop forgings", or if only part is forged then they are partly forgings and partly castings.

Washed Metal, cast iron from which most of the silicon and phosphorus have been removed by the Bell-Krupp process without removing much of the carbon, so that it still contains enough carbon to be classed as cast iron. The name "washed metal" is extended to cover this product even if its carbon is somewhat below the proper limit for cast iron[1].

Weld Iron, the same as wrought iron. Obsolescent and needless.

Weld Steel, iron containing sufficient carbon to be capable of hardening greatly by sudden cooling, and in addition slag-bearing because made by welding together pasty particles of metal in a bath of slag, as in puddling, and not later freed from that slag by melting. The term is rarely used.

White Pig Iron, and White Cast Iron, pig iron and cast iron in the fracture of which little or no graphite is visible, so that their fracture is silvery and white[2].

Wrought Iron, slag-bearing, malleable iron, which does not harden materially when suddenly cooled.

Wrought Steel, the same as weld steel. Rarely used.

[1] Unknown in Denmark, no equivalent, Hannover.

[2] Professor Wedding reports that the term "white pig iron" is applied in Germany to certain varieties of pig iron which, instead of being silvery and white, are dark. The president and secretary do not feel confident as to Professor Wedding's meaning.

III. Glossary of special sizes, shapes and conditions of iron and steel.

Names designating special sizes or shapes of iron and steel.

English	French	German	Swedish
Bar Iron, wrought iron in the form of bars, rods, etc.		Stabeisen	
Muck Bar, the rough bars usually 1 inch thick and about 4 inches wide, made by the first rolling of a ball of puddled iron.	Loupe. Lopin Ebauché de puddlage, Fer brut.		no equivalent
Merchant Bar, wrought iron in the form of merchantable bars or rods made by shearing muck bar into short lengths, piling it and rolling or forging it at a welding heat.			
Bloom 1. A large bar, drawn from an ingot or similar mass, for further manufacture. 2. A rough bar of wrought iron drawn from a Catalan or bloomary ball for further manufacture.	Bloom	Luppe Wolf	
Billett. A small bar drawn from a pile, bloom or ingot for further manufacture. The committee recommends that the line between blooms and billets be drawn at the size of 5 inches square[1], as representing common custom.	Billette	Packet	
Slab. A flat piece or plate, with its largest surfaces plane drawn or sheared from an ingot or like mass for further treatment.			

[1] Pourcel suggests 45 mm as a dividing line. — Brinell recommends 6″ square.

IV. The boundary between steel and iron.

It would be well to decide on a definite carbon-content to serve as a boundary line between ingot iron and ingot steel, between puddled iron and puddled steel, and between any other varieties of wrought iron and weld steel. Two plans have been considered. One is to draw this line at $0{\cdot}32\%$ carbon or its equivalent in other elements, for the reason that this carbon content appears to correspond to the critical point O in the diagrams of Roberts-Austen and Roozeboom[1]. This has the merit of corresponding to a definite physical boundary.

The other plan is to draw the boundary at $0{\cdot}20\%$ of carbon, because this is a convenient place to separate the important classes »soft steel« and »half hard steel« so that if this point was adopted »ingot iron« would be synonymous with »soft steel«, and »ingot steel« would be the equivalent of the two classes »half-hard steel« and »hard steel«.

Cambridge, Massachusetts, March 31st 1906.

[1] Mr. Pourcel would classify solely according to the presence or absence of slag, so that »steel« should include all forms of iron freed from slag by fusion and cast in a malleable condition, and »wrought iron« should include all classes produced in a pasty condition. He does not think that any cross classification according to the proportion of carbon is expedient.

Names						Apparatus in which usually made					
English	French	German	Swedish	Danish	Dutch	English	French	German	Swedish	Danish	Dutch
Species											
Cast iron	Fonte	Roheisen if not remelted, Gußeisen if remelted	Gjutjern	Stobejaern	Gietyzer (ruwyzer)	Blast furnace	Haut fourneau	Hochofen	Masugn	—	—
Varieties											
White cast iron or pig iron	Fonte blanche	Weißes Roheisen	Hvitt tackjern	Hvidtjaern	witgietyzer (witruwyzer)	Blast furnace	Haut fourneau	Hochofen	Masugn	—	—
Gray cast iron or pig iron	Fonte grise	Graues Roheisen	Grått tackjern	Graatjaern	grysyzer	Blast furnace	Haut fourneau	Hochofen	Masugn	—	—
Mottled cast iron or pig iron	Fonte truitée	--	Hagelstatt or Halfhwitt or halfgratt tackjern	Spragletjaern	wit korreligyzer	Blast furnace	Haut fourneau	Hochofen	Masugn	—	—
Pig iron (white, gray, mottled, etc.)	Gueuses de fonte ou fonte en gueuse	Gußeisen	Tackjern	Pigjaern or Raajaern	—	Blast furnace	Haut fourneau	Hochofen	Masugn	—	—
Hot metal. or direct metal	Fonte de première fusion?	Remeisen	direct gjutet fran Masugnen	Smeltet Raajaern	no equivalents	Blast furnace	Haut fourneau	Hochofen	Masugn	—	—
Basic cast iron or pig iron	—	—	—	—		Blast furnace	Haut fourneau	Hochofen	Masugn	—	—
Hematite cast iron or pig iron	—	—	—	—		Blast furnace	Haut fourneau	Hochofen	Masugn	—	—
Malleable pig iron	—	—	—	—		—	—	—	—	—	—
Washed metal	Fonte épurée	—	Tvattad	no equivalent		Pernot furnace	Four Pernot	—	Pernot's ugn	—	—
Refined cast iron	Fonte mazée	—	Raffineradtjern	Raffineret Raajaern		Coke refinery	Feu de finerie anglais	Feinherd or Feinofen	Koks raffineringsverk	—	—
Charcoal hearth cast iron	Fonte mazée	Herdfrischeisen	no equivalent	Raffineret Raajaern		Charcoal hearth	—	Frischherd	Fräkolshärd	—	—
Alloy cast iron	Fontes spéciales	—	Special tackjern	ne equivalent perhaps special raajaern		Blast furnace crucible or electric furnace	Haut fourneau four à creusets four électrique	—	Masugn, degel Elektrisk ugn	—	—
Species						Air furnace, open hearth furnace, or cupola furnace, followed by long annealing in boxes heated externally	Four à Réverbère four Siemens, Cubilot	—	Reverberugn, Martinsugn Kupolugn	—	—
Malleable castings	Fonte malléable	Schmiedbares Gußeisen or Schmiedbarer Guß	Aduceradtjern	Hammerbart Stöbejaern	—	—	—	—	—	—	—

Names						Apparatus in which usually made.					
English	French	German	Swedish	Danish	Dutch	English	French	German	Swedish	Danish	Dutch
Species											
Steel	Acier	Stahl	Stal	Staal	Staal	Bessemer converter, open hearth furnace or crucible furnace	Convertisseur Bessemer, four Siemens, four à creusets	Bessemer Birne, Flußeisen-Flammofen, Tiegelofen	Bessemer converter, martinsugn, degelstålsugn	--	—
Variety A.											
Called steel because cast initially into a malleable mass. 1. Soft or low carbon steel, or ingot iron	Acier doux, acier extra doux, fer fondu	Flußeisen [1]	Gôtmetall-	Staal Blodtstaal	Smeltyzer						
2. Half hard and hard, or medium and high carbon steel, or ingot steel	Acier fondu acier mi-dur acier dur	Flußstahl [1]	—	Haardtstaal	—	—	—	—	—	—	—
Sub-Varieties											
Bessemer steel	Acier Bessemer	Bessemer-Flußeisen Bessemer-Flußstahl	Bessemerstål	Bessemer staal	Bessemer staal	Bessemer converter	Convertisseur Bessemer	Bessemer Birne	Bessemer converter	—	—
Open hearth steel	Acier Martin Siemens, acier sur sole	Flammofen-Flußeisen Flammofen-Flußstahl	Martinstål	Martinstaal	Frischstaal	Open hearth furnace	Four Siemens	Flammofen (Martin- or Siemensofen)	Martinsugn	—	—
Crucible steel	Acier au creuset	Tiegelflußeisen Tiegelflußstahl	Degelstål or Degelgjutstål	Digelstaal	Kroerenstaal	Crucible furnace	Four à creusets	Tiegelofen	Degelstålsugn	—	—
Cast steel	Acier au creuset	Gußstahl	Gjutstål	Stobestaal	Gietstaal	Crucible furnace	Four à creusets	Tiegelofen	Degelstalsugn	—	—
Steel castings	Moulages d'acier	Flußwaren	Stålgjutgods	Staal-stôbegods, or Facongods	—	—	—	—	—	—	—
Variety B.											
Weld steel, or wrought steel called steel because it is capable of hardening greatly by sudden cooling	Fer fort ou fer dur	Schweißstahl or Schweiß-eisen [1]	Wäll stål rarely used, no equivalent	Svejsestaal	Welyzer	—	—	—	—	—	—
Sub-Varieties											
Blister steel, also called cemented and converted steel	acier poule, acier cémenté, acier de cémentation	—	Blåsstål Bräunstal, Cementstal,	Blaerestaal or Cementstaal	no equivalent cementstaal	Cementation furnace	Four de cémentation	—	Bräunstålsugn	—	—
Shear steel	Acier rafiné une fois corroyé	—	—	—	no equivalent	—	—	—	—	—	—
Puddled steel	Acier puddlé	Puddelstahl	Puddelstål	Puddelstaal	Puddelstaal	Puddling furnace	Four à Puddler	Puddelofen	Puddelugn	—	—
Variety C.											
Alloy steels	Alliages à base de fer, aciers spéciaux	Sonderstahl	Special stål	no equivalent perhaps Special staal	no equivalent	Bessemer converter, open hearth furnace or crucible furnace	Convertisseur Bessemer, four Siemens four à creusets	—	Bessemer converter martinsugn degelstal sugn	—	—
Species											
Wrought iron (or weld iron, or, in Great Britain, malleable iron)	Fer soudé	Schmiedeisen and Stabeisen	Smidesjern Lancashire jern	Svejsejaern	Welyzer	Puddling furnace	Four à Puddler	—	Puddelugn	—	—
Varieties											
Puddled iron	fer puddlé	Puddeleisen	Puddeljern	Puddeljaern	Puddelyzer	—	—	—	—	—	—
Bloomary or knobbled iron	Fer au bois (obtenu au Bas-Foyer)	—	Lancashire Franche-Comté or Walloon jern	Haerd frisket jaern (always refers to Lancashire iron from Sweden)	—	Bloomary, also loupe charcoal Lancashire knebblin and	Bas-Foyer, Feu d'affinerie	—	Lancashire-härd [2] Franche-Comté-härd Wallonhräd [3]	—	—

[1] According to Wedding cast metal having a tenacity greater than 50 kg per sq. mm should be called Flußstahl, while with a smaller tenacity it should be called Flußeisen. Weld metal with a tenacity exceeding 42 kg per sq. mm should be called Schweißstahl and with a less tenacity, Schweißeisen.

[2] Used in Dannemora district in Sweden exclusively for producing bars to be cemented and melted in crucible furnaces (Brinell).

[3] Fired with charcool and used in Sweden to a great extent (Brinell).

34 e.

INTERNATIONAL ASSOCIATION FOR TESTING MATERIALS

BRUSSELS CONGRESS 1906.

REPORT ON PROBLEM 34.

FIXING A UNIFORM DEFINITION AND NOMENCLATURE OF BITUMEN.

PRESENTED BY THE CHAIRMAN OF THE COMMISSION 34 PROFESSOR DR. G. LUNGE.

WITH AN APPENDIX

INVESTIGATIONS OF ASPHALT

BY G. LUNGE AND V. KŘEPELKA.

TRANSLATED BY E. ORMEROD, ZURICH.

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Fixing a uniform Definition and Nomenclature of Bitumen

by **Professor Dr. G. Lunge**, Zurich.

(Translated by E. Ormerod, Ph. D., M. Sc. Zürich.)

On November 5th 1902 the undersigned was notified by the President of the Association that the following gentlemen had been elected to sit on Commission No. 34:

Chairman:

Prof. Dr. G. Lunge, Polytechnikum, Zurich.

Vice-Chairman:

Dr. Jenő Kovács, Tataros (Post Mező-Telegd) Hungary (deceased).

Members:

Dr. M. Albrecht, Besitzer der Mineralwerke Albrecht & Co., Hamburg.

Dr. L. Eger, Munich, Arnulfstrasse 20, II.

Prof. Dr. D. Holde, Vorsteher der Abteilung für Öle am königl. Material-Prüfungsamt, Gr.-Lichterfelde.

Prof. Em. Paternò di Sessa, Senateur du Royaume d'Italie, professeur des applications de la chimie à l'Université de Rome.

L. Schmelck, Stadtchemiker, Christiania.

Dr. Max Böhm, Fabriksbesitzer, Přivoz b. M.-Ostrau.

Prof. B. Kirsch, k. k. technolog. Gewerbemuseum, Währingerstraße 59, Vienna IX.

Albert Grittner, Chef-Chemiker der kgl. ungar. Staatsbahnen, Budapest.

A. W. Dow, Inspector of Asphalts and Cements for the District of Columbia, Washington D. C.

Clifford Richardson, Asphalt Expert, New-York Testing Laboratory; Long Island City, N. Y.

A circular in German, French and English was immediately addressed to the various members (appendix No. 1), in which the undersigned expounded his working programme, and invited his colleagues to make him definite proposals, founded on their own investigations, for a definition and nomenclature of bitumen. He further requested to be informed of the different methods employed by them in analysing and testing the different kinds of bitumen, to enable him to draw up a system of classification founded on these investigations. In order that it would be possible for him to carry out the control analysis in his laboratory during the summer term he requested that the proposals be sent to him not later than May 1st 1903.

In October 1902 he, together with one of his students, Herr V. Křepelka, began an experimental work on the methods of analysing bitumen. This investigation, which was finished in the course of the summer 1903, is described more fully in appendix No. 4.

Unfortunately only very few answers were received by May 1st 1903; the undersigned therefore issued a second circular in three languages (appendix No. 2) requesting replies on or before November 1st, and finally a third one on November 20th (appendix No. 3) in which he again asked for reports as soon as possible.

It will be admitted that the undersigned did everything in his power to push forward the work entrusted to him, but the result attained was very small despite an extensive correspondence in several languages besides the aforementioned circulars. The following members of the commission have sent no answer whatever: Dr. M. Albrecht, Hamburg; Dr. Eger, Munich; Engineer Locherer, Paris; A. W. Dow, Washington. — Letters with no important information were received from: Prof. Dr. Holde, Charlottenburg; L. Schmelck, Christiania, who excused themselves on the plea of ill-health, further from A. Grittner, Budapest; Prof. Kirsch, Vienna; Prof. Paternò, Rome. — Mr. Richardson, Long Island City, wrote me fully and gave me detailed information regarding

American asphalt and the methods used by him in analysing the same. This gentleman endeavoured to get together an American committee, but without success, as the persons concerned took too little interest in the question. In his last letter dated November 10th 1903 he says, he had prepared between three and four hundred pages of Manuscript on this subject, but feeling that his results were not such as could be placed before our commission he declined to make any report.

I received only two opinions on this subject of any importance to our commission, one from Dr. Jenő Kovács, Tataros (Hungary), and the other from Dr. Max Böhm, Přívoz near Mährisch-Ostrau.

Dr. Kovács states that he understands under «asphalt» only those materials consisting of, or containing asphalt-bitumen, the bitumen of which has resulted from natural resinification or oxidation. The materials, which he divides into the following classes: asphalt-bitumen, asphalt-rock, asphalt-sand, asphalt-mastic, liquid asphalt (asphalte coulé), compressed-asphalt (asphalte comprimé), insulating asphalt and asphaltic roofing paste, ought to be characterised by the following properties: they are elastic, do not soften at 40—50° C. or split or crack at low temperatures (—15° to —20° C.). They contain bodies which volatilize with difficulty and do not oxidise at the ordinary temperature, and are therefore least exposed to decay through atmospheric influences. Pure bitumen is of a bright black colour with a reddish shimmer and forms either a viscous, sticky or else a hard and brittle mass which is not fluid under 35° C. It dissolves completely in carbon disulphide, oil of turpentine and chloroform, partly in petroleumspirit and benzol and is almost insoluble in alkohol. By the name «artificial-asphalt», or more correctly «asphalt-surrogate» he designates those materials and products the chief component of which is not asphalt-bitumen, but coal-tar-pitch, petroleum-pitch (according to him wrongly called petroleum-asphalt or bitumen) or other similar resinous products which cannot be discriminated outwardly from the natural products, but whose inferiority or uselessness shows itself in a short time because the binding substance does not possess the necessary physical and chemical properties.

Dr. Kovács accordingly limits the term «asphalt» to the genuine natural-asphalt, and will not hear of it being applied to the products from coal-tar or petroleum.

Dr. Böhm also limits the term «asphalt» exclusively to the natural product, but describes the residues remaining after the distillation of petroleum, lignite-tar, coal-tar and fatty acids according to their degree of fluidity as «pitch» or «tar» with a prefix showing the origin of the product. For instance in the case of pitch: coal-pitch, lignite-pitch, petroleum-pitch, stearine-pitch, wool-fat-pitch; in the case of tar: coal-tar, lignite-tar, petroleum-tar. The word «Goudron» may be used instead of «tar», and «bitumen» should serve as a general term for all the abovementioned solid or liquid, natural or artificial hydrocarbons.

Dr. Böhm agrees so far with Dr. Kovács that not only the artificial bitumen produced from coal-tar, but also that derived from petroleum ought not to be described as «asphalt». He does not, however, say how petroleum-pitch may be distinguished analytically or physically from natural-asphalt. Mr. Grittner informs me, he has not been able to make such a distinction and I have also come to the same conclusion after the exhaustive series of experiments made together with Mr. Křepelka which are described in the appendix. The tenor of Mr. Clifford Richardson's report amounts to the same. His argument is, that the formation of genuine natural-asphalt has been very similar to that of petroleum and that various transition stages exist between them, which easily explains why bitumen produced artificially from petroleum cannot be distinguished by any particular characteristic from natural-asphalt.

For the same reason and also on the basis of my own investigations I consider that the term «asphalt» should also be applied to products made from petroleum, but that a distinction should be made between «natural-asphalt» and «petroleum-asphalt» («Erdöl-Asphalt»). On the other hand I quite agree that the products made from coal-tar (and lignite-tar) and pitch can be distinguished from the asphalts, not only in practical use, where they exhibit their inferiority, but also by laboratory tests, and should be classed as pitch or tar and not as asphalt. I cannot, however, extend this prohibition to the products made from petroleum, firstly, because these products are genetically similar, secondly, because they cannot be distinguished from one another with any degree of certainty in the laboratory and thirdly because I have no positive proofs that petroleum-asphalt, in reference to its technical uses, cannot hold its own in competing with the numerous kinds of natural-asphalt.

Above all I should certainly say that the term «natural-asphalt» ought never to be used for the products artificially prepared from petroleum.

Unfortunately the number of expert opinions received on this subject is so small that none of them could be proposed as conclusive.

In conclusion it may be mentioned that a monograph on all the different kinds of asphalt will very shortly (in any case before the congress) be published by the well known specialist Dr. H. Köhler.*)

Zurich, January 5th 1904.

G. Lunge.

*) This work has in the meantime appeared under the title: »Chemie und Technologie der natürlichen und künstlichen Asphalte« (Brunswick 1904). From this I see that this expert is quite of the same opinion as myself as to the classification of bitumens. According to him the restriction of the term »asphalt» to natural-asphalts, demanded by Kovács, cannot be recognised, because there is no reason for separating the asphaltic products, prepared by the distillation and further treatment of petroleum, from natural-asphalt to which they are very similar, if not identical. There is also no reason why these products should be classed with the tars, which are formed by the pyrogenic decomposition of fossil fuel and are entirely different in their physical properties from the asphalts.

June 1905. G. Lunge.

Appendix I.

Zurich, November 1902.

Mr. . . . . .

Member of Commission 34 of the International Association for testing Technical Materials, for fixing a uniform definition and nomenclature of bitumen.

Dear Sir,

I have been informed by the president of the Association that you have had the kindness to promise your cooperation to Commission 34, and I, as chairman of this Commission have been instructed to make arrangements with the members about their share of the work.

In the first place I beg to ask what share of the work you are in a position to undertake for the Commission. Definite propositions for the definition and nomenclature of the materials concerned should be made, based either on experience with a large number of the different kinds of bitumen met with in trade, or investigations in the laboratory. The undersigned, possibly with the help of other members of the Commission, will collect these propositions and prepare a report on the same, which will be laid before the next Congress. The methods for analysing bitumen adopted by the members will, as far as possible, be re-examined in the laboratory of the undersigned.

The principal point is to distinguish with certainty between the natural bitumens and asphalts and those artificially prepared from coal-tar and petroleum-residues. The Commission will, however, make it their duty not only to fix definitions but also to classify the methods for investigating and examining the materials in question.

I therefore request you to let me have your report not later than May 1st 1903. It will then be possible to compare the different methods of analysis in my laboratory during the

summer of 1903, and to prepare the above mentioned report, which must be in the hands of the president not later than April 1st 1904.

Kindly let me have the reply to the questions mentioned at the beginning as early as possible. You would also greatly oblige by sending me a large authentic sample (about 1 Kilo) of any natural or artificial asphalt or bitumen in your possession, together with full particulars of its origin and its present trade-name, so that I may be able to use them for the analyses now being done in my laboratory.

Yours truly,

Dr. G. Lunge
Professor at the eidg. Polytechnikum, Zurich.

Appendix II.

Zurich, May 8th 1903.

Mr.

Member of Commission 34 of the International Association for testing Technical Materials.

Dear Sir,

In my circular of November 1902 I requested the members of the Commission to let me have their propositions on the definition and nomenclature of natural and artificial asphalt and bitumen, together with their methods of analysing the same, on or before May 1st 1903. So far I have received very few reports. As far as the proposals for the definition and nomenclature are concerned, the time can be extended. I must, however, request you to let me have them at the latest by November 1st 1903, since the Board of Directors must have the reports of the Commission by the end of the year in order that they may be printed in time for the St. Petersburg Congress. Moreover the drawing up of the report by the undersigned will naturally require time.

Yours truly,

G. Lunge.

Appendix III.

To the Members of Commission 34 of the International Association for testing Technical Materials.

Dear Sir,

In my circular of May 8th last I requested you to let me have your proposals for the definition and nomenclature of natural and artificial asphalt and bitumen not later than November 1st, as I am obliged to hand in my report to the Board of Directors by the end of the year.

I have heard nothing further from you, and again repeat my request for an early report.

Yours truly,

G. Lunge.

Appendix IV.

Investigations of Asphalt.

By G. Lunge and V. Křepelka.

The appointment of Commission No. 34 of the meeting of the International Association for testing Technical Materials in Budapest, September 1901, charged with the establishment of a uniform definition and nomenclature of bitumen, gave rise to the investigation of asphalt described below.

The chairmanship had been entrusted to one of us (G. L.) whose duty it was to obtain the opinions of the other members in order that they might be laid before the next Congress at St. Petersburg. He did not, however, think he ought to be satisfied with this formal participation, but considered it his special duty to study closely the properties of these bodies as a necessary basis for a rational nomenclature of asphalt and bitumen, taking particular notice of the distinguishing properties by which the individual members of the different classes, according to their origin, could be distinguished with sufficient certainty for practical purposes. There is no scarcity of publications on this subject, as may be seen from the list given below. In the first place the manifold and extensive analyses of Clifford Richardson deserve special mention. But the authors of those treatises will be the first to admit that very much still remains to be done in this direction, and the authors of this report naturally do not imagine they have solved more than a very small part of the whole problem. Nevertheless they hope that their work has enabled them to add a contribution to the investigation of this subject.

The advice and supervision of the work were supplied by one of us (G. L.), whilst the practical part was carried out by the other (V. K.). We are indebted to the following gentlemen and firms for the material for our work: Dr. Jenő Kovács, Tataros (Hungary); Clifford Richardson, Long Island City, New York; Prof. Dr. D. Holde, Charlottenburg, and theVal de Travers Asphalt Company. As special literature for our purpose the following pamphlets were sent us by the respective authors.

Dr. Jenő Kovács. «Über Asphalt, sein Vorkommen, seine Verwendung und einschlägige Untersuchung», Budapest 1901 (from the debates of the Budapest Congress of the International Association for testing Technical Materials).

Asphaltum. The Philadelphia Museum Scientific Department. Bulletin No. 2.

J. W. Howard. «Natural-Asphaltum and its compounds.» Troy, New York 1894.

Clifford Richardson. «On the Nature and Origin of Asphalt.» New York 1898.

ditto. The Determination of Paraffin in Petroleum Residues, Asphaltic Oils etc. (Journ. Soc. Chem. Ind. May 1902), and also a long written report by the same author.

These works contain excellent descriptions of the occurrence and uses of asphalt as well as the scientific and techno-analytical investigations made up to now, and anyone who wishes to study the subject further should refer to them.

The asphalts or asphaltic materials analysed by us were the following (selected from the large number of samples placed at our disposal by the above named gentlemen).

I. 1. From Limmer, containing according to J. Kovács: 13·4% bitumen, 67% calcium carbonate, 19·5% ferric oxide and clay.

2. From Lobsann, containing according to L. Malo*): 11·9% bitumen, 69·6% calcium carbonate, 0·3% magnesium carbonate, 3·65% sand, 5·6% sulphur, 4·45% iron sulphide, 5·5% other substances.

3. From Val de Travers, according to L. Malo: 0·5% water and gases, 10·10% bitumen, 87·95% calcium carbonate, 1·45% other substances.

4. From Tataros, according to J. Kovács 15—22% viscous liquid asphalt, 78—85% sand.

*) L. Malo's analyses are quoted according to Kovács, to whom I am also indebted for the materials.

5. From Bermuda (Venezuela) according to Sam. P. Sadtler, 90% bitumen, and 10% foreign inorganic and organic constituents.
6. From Seyssel, according to L. Malo: 1·9% water and gases, 8·0% bitumen, 89·55% limestone, 0·55% other substances.
7. From Trinidad (unknown whether lake or land pitch).
8. From Selenico (no details given).
9. Gilsonite from Utah.

II. Artificial asphalts prepared from petroleum-pitch.

1. From Galician petroleum.
2. » petroleum-pitch, origin unknown.
3. » Wietze petroleum.
4. » Beaumont oil (Texas).

III. Coal-tar-products.

1. Soft pitch.
2. Hard pitch.

The origin of all these materials can (with the exception of II, 2) be regarded as quite certain, and they therefore formed a reliable basis for our experiments.

It was our first endeavour, as a basis for a practical method of distinguishing these products from one another, to discover a solvent which at the same time should be suitable, and easily obtainable in a pure condition. The solvents which have long been used for this purpose were investigated as to their effects. We tested more especially the different naphthas, oil of turpentine, benzol and chloroform. The extractions were always made in a Soxhlet apparatus. The solvent which proved to be the most suitable for our purpose was chloroform which was used in the form of the technically pure product (Kahlbaum). The difference in effect observed by Cl. Richardson between chloroform containing a little water and absolute chloroform was not noticeable in our experiments.

The raw bitumen was separated from the mineral and insoluble organic constituents by extraction with this solvent and the extract heated for a short time in an air bath at 120° C. to drive off any

remaining traces of chloroform. The pure bitumen was then investigated in the following directions.

A) Specific gravity
B) Melting point
C) Specific gravity of the chloroform solution
D) Iodine value.

No experiments were made in the direction of fractional solution, fractional distillation, analysis by combustion etc. etc., partly because only in a few cases had we sufficient material, and partly because the investigation of such a number of substances would have taken too long. As we learnt from experience a really useful classification of the various materials in this manner would have offered too many difficulties for technical as well as for scientific purposes.

A. Specific Gravity of Bitumen.

To remove any enclosed air-bubles the samples were melted, transferred to a Sprengel's pyknometer and then the determination carried out as usual. All determinations were made at 17° C. The results given are in each case the average of 3 determinations.

I. Natural asphalts.		II. Petroleum asphalts.	
1. Limmer . . .	0·9877	1. Galician . . .	1·0365
2. Lobsann . .	1·0342	2. Unknown . .	1·1075
3. Val de Travers	1·0832	3. Wietze . . .	1·1269
4. Tataros . . .	1·0931	4. Beaumont . .	1·1741
5. Bermudas . .	1·0861		
6. Seyssel . . .	1·0762	III. Tar-pitches.	
7. Trinidad . .	1·0994	1. Soft pitch . .	1·2365
8. Selenico . .	1·1131	2. Hard pitch . .	1·2741
9. Gilsonite . .	1·1495		

Leaving the remarkably low specific gravity of the Limmer-asphalt out of the question, both the natural and petroleum-asphalts vary between 1·03 and 1·15; only the Beaumont-asphalt (II. 4) being a little higher viz. 1·17. Natural and petroleum-asphalts cannot therefore be distinguished from one another by this method.

but the pitches, even soft pitch (the latter 1·2365) are so much heavier that no confusion is possible, and they may easily be distinguished from the rest by means of the specific gravity of the bitumen.

B. Melting point.

The determination of the melting point of pitch, asphalt etc. was greatly facilitated by the apparatus described by Kraemer & Sarnow (Chem. Ind. 1903, page 55), which we used throughout our investigation. We did not, it is true, succeed in obtaining the exactness mentioned in their paper, despite a strict observance of all the conditions according to these authors any error of observation should be nil or confined to a fraction of a degree. In four tubes charged identically and with the same class of bitumen, slowly heated in the same beaker, variations in the melting point of 1^0—2^0, and at times even 3^0—4^0, were often observed; but on making a greater number of experiments more certainty was obtained than with any of the other methods employed in analysing these substances.

The following figures are in each case the average of 4 determinations made with a corrected thermometer.

I. Natural asphalts.		II. Petroleum asphalts.	
1. Limmer	16·0° C.	1. Galician	18·2° C.
2. Lobsann	25·0° „	2. Unknown	37·8° „
3. Val de Travers	28·1° „	3. Wietze	118·2° „
4. Tataros	33·6° „	4. Beaumont	69·3° „
5. Bermudas	51·1° „	III. Tar-pitches.	
6. Seyssel	53·1° „	1. Soft pitch, raw	36·1° „
7. Trinidad	55·2° „	Extract of same	20·0° „
8. Selenico	90·0° „	2. Hard pitch, raw	113·0° „
9. Gilsonite	123·0° „	Extract of same	23·0° „

These figures show neither a differentiation of the various kinds of Bitumen, nor a regular relation between the specific gravity and the melting point. In many cases, however, high specific gravities correspond to high melting points.

C. Specific Gravity of the Chloroform Solutions.

It was from the outset to be expected that the varying composition of bitumen, although very little is known either qualitatively or quantitatively on this subject, would make itself conspicuous by the fact that solutions of the various kinds of bitumen of equal percentage would possess different specific gravities. It was therefore possible that a distinction between the different classes of asphalt might be arrived at by this means. This involved experiments on a large scale which had to be limited to that solvent which was recognised earlier as the most suitable, viz. pure chloroform. These experiments demonstrated that on dissolving the bitumen in chloroform in no case was there an increase but always a decrease in the specific gravity of the solvent, i. e. a dilatation, so that the product was more like the mixture of two fluids than the usual solution of a solid in a liquid, which fact is, however, easily explained by the amorphous semi-liquid character of bitumen.

The determinations were carried out as follows:

A chloroform solution containing about 6% was prepared from a carefully weighed out quantity of bitumen in a stoppered graduated flask and the solution cooled to the temperature of the room. A Sprengel's pyknometer was filled with the solution, the corrected thermometer inserted, the outside of the apparatus cleaned with chloroform, dried and the temperature allowed to rise to 17·5°, the liquid flowing from the capillaries being removed. This heating process was at first done in a water bath, which, of course necessitated very careful drying of the apparatus afterwards. Later on on equally good results were obtained in a much easier manner by hanging up the pyknometer by means of a piece of thread in the balance room (the temperature of which was at least 17·5°, usually a little higher) and swinging it backwards and forwards till the above temperature was reached, when the capillary tubes were closed and the pyknometer quickly weighed. Each determination was repeated at least twice. After this the flask in which the bitumen had been dissolved (which was well stoppered) was again weighed and the quantity of bitumen still present in the solution determined by calculation. A certain quantity of fresh chloroform was then added making the solution about 5%, the specific gravity of the

same being again determined. In this way solutions of varying concentration were prepared and investigated. Owing to the fact that chloroform evaporates very rapidly it is necessary to work with the utmost despatch, otherwise, in spite of the apparatus being well closed, errors will be caused by the evaporation of the solvent. The chloroform used was Kahlbaum's special rectified of specific gravity 1·4915 at 17·5°. The pyknometer had a capacity of 18·2695 gr water at 17·5°.

The following figures give the specific gravities at $\frac{17{\cdot}5^0}{17{\cdot}5^0}$ the same being determined by weighing in air without further correction. The actual figures obtained were as follows (average of at least two determinations).

I. Natural asphalts.	Percentage of the Chloroform solution	Specific gravity
1. Limmer.	5·78	1·4499
	4·58	1·4573
	2·63	1·4695
	1·94	1·4751
	0·91	1·4837
2. Lobsann.	6·02	1·4653
	3·95	1·4743
	1·44	1·4850
3. Val de Travers.	7·52	1·4573
	4·37	1·4696
	3·75	1·4735
	3·07	1·4765
	2·42	1·4798
	2·10	1·4810
	1·61	1·4844
	1·21	1·4861
	0·92	1·4873
4. Tataros.	5·72	1·4617
	4·83	1·4660
	3·49	1·4731
	3·00	1·4756
	1·66	1·4822

5. Bermudas.	5·72	1·4608
	4·83	1·4659
	2·95	1·4758
6. Seyssel.	5·77	1·4552
	3·74	1·4685
7. Trinidad.	6·18	1·4589
	5·82	1·4613
	4·02	1·4680
	2·81	1·4762
	2·34	1·4790
	1·37	1 4840
	0·68	1·4872
8. Selenico.	5·61	1·4664
	4·58	1·4706
	3·66	1·4746
	2·75	1·4790
9. Gilsonite.	5·56	1·4646
	3·53	1·4759
	2·53	1·4805
II. Petroleum asphalts.		
1. Galician.	5·62	1·4561
	4·67	1·4601
	3·29	1·4687
	2·67	1·4736
	1·67	1·4795
	0·78	1·4849
2. Unknown.	6·20	1·4558
	4·27	1·4633
3. Wietze.	7·40	1·4632
	5·88	1·4699
	4·01	1·4766
	2·96	1·4804
	1·83	1·4851
	0·65	1·4879
4. Beaumont.	6·15	1·4611
	5·17	1·4660
	3·41	1·4746
III. Coal-tar-pitches.		
1. Soft-pitch.	5·96	1·4752
	4·00	1·4808
	2·83	1·4842

2. Hard-pitch.	5·53	1·4803
	5·50	1·4816
	4·41	1·4835
	3·67	1·4850
	2·84	1·4861
	1·98	1·4879
	1·14	1·4896
	0·70	1·4905
	0·31	1·4910

From the above data curves were constructed, which, however, are not reproduced here, as the scale required to keep the curves separate and distinct from one another is so large that too much space would be required. We shall, however, give the conclusions arrived at from a study of the original. In almost all cases is the line joining the separate points for each bitumen a straight one or only slightly curved and there is unquestionably a distinct and regular connection between the quantities dissolved in the chloroform and the dilatation of the solvent caused thereby. From these curves it is possible in each case to calculate the quantity of bitumen contained in the solution from the specific gravity, and to found thereon an analytical method for the valuation of the raw material. But this method is not capable of general application, because the influence of each separate bitumen on the specific gravity of the solution is different from the rest. No two curves ever coincided exactly and it is quite certain that no two of the investigated asphalts so far agreed in their composition as to be capable of being analysed quantitatively by one general physical method. Still the curves are arranged in groups which allow the different classes of bitumen to be distinguished from one another with absolute certainty.

Quite a distinct difference is at once noticeable in the curves representing the chloroform solutions of the coal-tar-pitches and those of the rest of the bitumens, corresponding with the aforementioned differences in the specific gravities. Taking the percentage of bitumen as abscissa and the specific gravity as ordinates (commencing with 1·4915 for pure chloroform), the curves for the tar-pitches do not rise so high, that is to say the dilatation is much smaller. As might be expected the lowest curve is that for hard pitch and immediately above this comes soft pitch. Next above this, the distance above the curve for soft pitch being about equal to

the distance between hard pitch and soft pitch, comes the asphalt from the Wietze petroleum and then at an equal distance above this begins a group of curves lying very close together representing all the natural asphalts with the exception of that from Limmer, but containing in their midst the petroleum asphalt from Beaumont. Separated from these by a very distinct interval appears the group of curves for the two petroleum asphalts (from Galicia and that from of unknown origin) and the asphalt from Seyssel. Finally at the very top and again separated by a distinct interval comes the curve for the natural asphalt from Limmer, which in this as in all other respects takes up quite an abnormal position.

That the chloroform curve for the Beaumont petroleum-asphalt appears in the middle of the natural asphalts is explained by the fact that, according to Richardson, the Beaumont oil occupies quite a singular position amongst the petroleums.

The above mentioned relations may be seen almost as distinctly from the following figures as from the curves. These figures are derived from the specific gravity curves of solutions of exactly 6% of bitumen in chloroform, which in some cases necessitated slight interpolations. They are arranged in order of ascending specific gravities, the difference in the two last decimals being given, as well as the specific gravity and melting point of the bitumen itself.

	Specific gravity of the bitumen	Melting point of the bitumen C.	Specific gravity of a 6% solution in chloroform	Difference
Limmer	0·9877	16·0°	1·4485	0·0054
Seyssel	1·0762	53·1°	1·4539	6
Gal. petroleum-asphalt	1·0365	18·2°	1·4545	20
Unknown. petroleum-asphalt	1·1075	37·8°	1·4565	25
Trinidad	1·0994	55·2°	1·4590	2
Tataros	1·0931	33·6°	1·4592	3
Bermudas	1·0861	51·1°	1·4595	24
Beaumont petroleum asphalt	1·1741	69·3°	1·4619	2
Gilsonite	1·1495	123·0°	1·4621	11
Val de Travers	1·0832	28·1°	1·4638	15
Selenico	1·1131	90·0°	1·4648	5
Lobsann	1·0342	25·0°	1·4653	40
Wietze petroleum-asphalt	1·1269	118·2°	1·4693	58
Soft coal-tar-pitch	1·2365	36·1°	1·4751	51
Hard coal-tar-pitch	1·2741	113·0°	1·4802	

From this list it will be seen that there is absolutely no relation between the melting point and the specific gravity of the chloroform solution and only in a few cases can any relation between the specific gravities of the bitumens and those of their chloroform solutions be observed. It is therefore not possible by this means to distinguish the genuine natural asphalts from the petroleum-asphalts and still less is it possible to draw distinctions between the individual members of the same group, which would give any proof of their origin; the only exception being the extreme cases of Limmer and Wietze.

D. Iodine values.

As is well known Hübl first introduced the „iodine value" as a means for distinguishing fat oils from other oils of the same class, that is to say the quantity of iodine used up from a mixture of iodine and mercuric chloride (which serves as an iodine carrier) in alcoholic solution, the same being estimated by titrating back with sodium thiosulphate. It was plausible that this method would be applicable to our purpose. Here as in the case of the fat oils the iodine is bound by being added on to the unsaturated valencies. The reactions which thereby take place are however rather complicated and the combination of the iodine with other than unsaturated bodies in the form of substitution products is, in certain cases, not impossible[1]). It is therefore necessary always to work under the same conditions. The determinations were carried out as follows.

Solutions containig 25 gr pure iodine in 500 c.c. 95% alkohol and 30 grm. mercuric chloride in the same quantity of alkohol were prepared and mixed in equal proportions two days before use. 25 c.c. of this solution were then added toa solution of 0·3 grm. bitumen in 20 c.c. chloroform, and the mixture allowed to stand for 24 hours at the ordinary temperature, protected from the direct rays of the sun. Further 20 c.c. chloroform and 25 c.c. mercuric chloride solution without bitumen were treated in exactly the same manner and allowed to stand for 24 hours. Then 20 c.c. of a 10%

[1]) In our experiments such a combination can scarcely have taken place no formation of hydriodic acid having been observed.

potassium iodide solution and 300 c c. water were added to the contents of each bottle and both titrated with $^1/_{10}$ N sodium thiosulphate solution (final titration). A mixture of chloroform, mercuric chloride solution, potassium iodide and water prepared in exactly the same manner was titrated immediately (first titration). The iodine value was calculated from the mean of the first and final titrations.

These directions are taken from Fr. M. Kitt's paper «Die Jodzahl der Fette und Wachsarten». The method described by Hanuš (Zeitschr. f. Unters. d. Nahrungs- u. Genußmittel 1901) was also tried. By this method the iodine number is determined with a solution of iodine bromide in glacial acetic acid, whereby much time is saved, but the figures obtained are lower than those given by Hübl's method, further the differences between the various kinds of bitumen varied so considerably that this method was not used by us for the comparison of bitumens.

The following figures were obtained as the mean of two determinations (which nearly coincided) carried out according to Hübl's method.

I. Natural asphalts.		II. Petroleum asphalts.	
1. Limmer	23·37	1. Galician	44·79
2. Lobsann	23·84	2. Unknown	44·26
3. Val de Travers	26·53	3. Wietze	52·02
4. Tataros	39·71	4. Beaumont	46·97
5. Bermudas	46·28		
6. Seyssel	44·33		
7. Trinidad	54·10	III. Coal-tar-pitches.	
8. Selenico	46·99	1. Soft	69·34
9. Gilsonite	46·68	2. Hard	64·39

A relation between the iodine values and those given under the headings A, B and C, i. e. melting point, specific gravity and specific gravity of a chloroform solution, is not apparent, as a comparison with the above figures proves. We can therefore only use them for characterising the chief classes and not, as in the case of solid oils etc., to form conclusions as to the presence of a particular kind of asphalt.

It may be mentioned that Cl. Richardson, who investigated the bromine absorption of a series of asphalts, was not able, from the figures he obtained, to distinguish between the various kinds.

Conclusions:

The experiments herewith described have resulted in the addition of some new quantitative characteristics to the already well known qualitative methods of distinguishing between coal-tar-pitch and natural asphalt on the one hand, and artificial asphalts made from petroleum on the other. In the first place the specific gravities of the extracted bitumens differ greatly for the two classes. The asphalts (natural and artificial) seldom rise above 1·1, only one, the Gilsonite asphalt being 1·17, whilst tar-pitch is considerably over 1·2. The chloroform solutions also show similar differences. The asphalts give wtih chloroform a considerably greater dilatation than tar-pitch, for a 6% solution even in the extreme case of the Wietze petroleum-asphalt the specific gravity is not quite 1·47, whilst in the case of the tar pitches it is 1·475 or more. Thirdly a distinction can be made by means of the iodine values, which for the asphalts are mostly under 50, rising to a maximum of 54, whilst for the tar-pitches they vary between 64 and 69.

To the already well known qualitative methods of distinction the following may be added:

On the addition of petroleum-spirit to a chloroform solution of coal-tar-pitch a precipitate is formed which is not the case with natural or petroleum-asphalts.

It was not, however, found possible to distinguish quantitatively between «natural asphalts» and «petroleum asphalts» obtained from petroleum. These two classes of products are apparently too closely related to one another and under the investigated conditions the figures obtained for the individual members appeared to be quite arbitrary.

This can scarcely be wondered at as it is a generally accepted view that the «asphalts» (semi solid or solid «natural asphalts») found in nature mixed with more or less mineral and other impurities, stand in very close relationship to the more or less liquid petroleums and are formed from these by analogous processes to those by which «petroleum asphalt» is prepared artificially from the oils.

Zürich, November 1903.

Supplement to the Report of Commission 34 of the International Association for testing Technical Materials.

Fixing a uniform Definition and Nomenclature of Bitumen.

As mentioned in the main report, at that time only very few of the opinions of the members of Commission 34 had been received. After a lengthy correspondence in the course of this year I have succeeded in getting two more important opinions, extracts of which follow:

Prof. Dr. Holde of Berlin wishes the term «natural asphalt» to be reserved exclusively for the natural black, tough bitumen which has up to now been called «asphalt», and he calls the bitumen produced artificially by the distillation of asphalt sand etc. (for instance in Tatáros) and the products obtained by the distillation of petroleum «artificial asphalt». He arrives at the following conclusions:

1. The term «asphalt» should be used to designate natural asphalt, which is almost entirely soluble in benzol, contains considerable quantities of sulphur (1·5—10 %) and is employed for building purposes, for protecting articles from dampness, for the manufacture of varnishes etc.

2. Those substances artificially obtained as residues by the distillation of mineral-tar, coal-tar, wood-tar, lignite-tar, petroleum and shale-oil, which are in appearance and also to some extent in their chemical and physical properties similar to natural asphalt and may be used as a substitute for this, should most certainly be marked in such a way by those persons or firms bringing the asphalt into the market, that they may be easily distinguished from natural asphalt. This demand appears to be imperative and ought eventually to be supplied by suitable arrangements, because on the one hand the investigation as to the origin is very difficult (in practice is found in the majority of cases to be impossible) and on the other hand opinions differ very widely as to the suitability for technical purposes of the various natural and artificial asphalts.

It would perhaps be as well to class the artificial asphalts according to their origin as follows:

1. Artificial asphalt (petroleum pitch or petroleum asphalt)
2. „ „ (coal-tar-pitch)
3. „ „ (lignite-tar-pitch)
4. „ „ from soft natural asphalt bitumen etc.

Mr. S. F. Peckham, city analyst for New York, proposes definite names for the different kinds of bitumen, of which he recognises thirty. He divides these thirty substances in the following three classes.

A) Pure bitumen as found in nature.

B) Pyro-bitumen which when heated gives substances more or less similar to natural bitumen.

C) Artificial products obtained by the distillation or chemical treatment of pyro-bitumen and other crude materials. These should be distinguished as «artificial bitumen».

To class A (bitumen proper) belong:

1. Natural gas
2. Natural naphtha, quite or almost colourless and easily volatile.
3. Petroleum or naphtha
4. Maltha or mineral tar
5. Petrified paraffin (ozokerite)
6. Asphalt coal which on distillation gives paraffin (Grahamite, Albertite, Gilsonite).
7. Asphalt from the Dead Sea (shining pitch) which on distillation does not give paraffin, but other hydrocarbons.
8. Mineral tar mixed with limestone from Seyssel etc.
9. „ „ „ „ sandstone from various districts
10. „ „ „ „ sand from India
11. „ „ „ „ earthy matter from Lake Trinidad etc.

To class B belong:

12. Peat
13. Lignite
14. Bituminous coal
15. Semi bituminous coal

16. Anthracite
17. Boghead and other bituminous paraffin shales
18. Bituminous shale from Autun etc.

Other experts do not consider this class as belonging to the «bitumens».

Class C comprises all those substances obtained from those in classes A and B by distillation at low or high temperature or by chemical treatment

19. Coal gas (illuminating gas)
20. Refined petroleum (illuminating petroleum)
21. Lubricating oil from petroleum etc.
22. Paraffin, Ceresin
23. Coal-tar, furnace tar, coke oven tar
24. Residue oil from petroleum
25. Pittsburg «Flux», «Dubbs Asphalt», Bylerite
26. Solid residue from Californian or Texas petroleum
27. Coke-pitch, brittle residue from petroleum, coal-tar-pitch
28. Acid tar and acid pitch
29. Candle-tar.

As will be seen, Peckham enumerates a number of substances which do not come under the scope of Commission 34. His classification, which in itself is highly interesting, will consequently scarcely be suitable for our purpose.

December 22nd 1904.

Prof. Dr. Lunge.
Chairmann of Commission 34.

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INTERNATIONAL ASSOCIATION FOR
TESTING MATERIALS.

BRUSSELS CONGRESS 1906.
REPORT ON PROBLEM 35.

Group L

STUDY OF THE METHODS OF TESTING CAOUTCHOUC.

REPORT PRESENTED WITH

NOTES ON THE ANALYSIS OF MANUFACTURED RUBBER.

BY **EM. CAMERMAN**, BRUSSELS,
CHAIRMAN OF THE COMMISSION 35.

Study of the methods of testing caoutchouc.

(Decision of the Budapest Congress 1901.)

Report presented by **E. Camerman**, President of Commission 35.

(Translated by F. H. Leeds, F. I. C., London.)

The Commission appointed for the solution of Problem 35 consisted of following members:

E. Camerman, Brüssels, Chairman.
Englebert, Liège, Belgium.
A. Jacobsen, Copenhagen, Denmark.
Dr. Eger, Munich, Germany.
P. Breuil, Paris, France.
G. B. Pirelli, Milan, Italy.
E. Simonsen, Christiania, Norway.
A. v. Boschan, B. Kirsch, } Vienna, Austria.
G. Heyse (†), B. Kempe, } St. Petersburg, Russia.
F. Maly, Budapest, Hungary.
R. S. Pearson, New-York, U. S. A

The only important paper to be mentioned by the President is the memoire prepared by M. Breuil, Departmental Chief in the „Laboratoire du Conservatoire des Arts et Métiers", Paris, which

deals with the mechanical testing of caoutchouc, and is full of valuable information. Unfortunately, this paper has been found too lengthy for a complete reprint, only an abstract has been published as „non-official paper", and the reader who is interested in the matter should therefore consult the columns of the journal „Le Caoutchouc et la Gutta-Percha" (No. 1, March 15th, 1904, et seq.)* where it has appeared in extenso.

The object of the following note is to refresh the reader's memory of what is most trustworthy in our existing state of knowledge. Methods of analysis that seem to have been proved accurate are described; and the whole paper may be regarded as a basis for future investigation, for improvements in our present processes, and for the further work to which the next Committee will devote its attention.

* Published at 49, Rue des Vinaigriers, Paris.

Notes on the analysis of manufactured rubber.

By **E. Camerman**, Engineer, and President of Committee 35.

Caoutchouc is a hydrocarbon of the terpene series, the generic formula of which is $C_{10}H_{16}$. The terpenes are unsaturated hydrocarbons, and as such are able to combine with atoms of chlorine, bromine, iodine, oxygen, or sulphur.

Caoutchouc is a substance that exudes from trees or sarmentose plants of numerous kinds and species. The plants do not all yield the hydrocarbon, but rather a mixture of terpenes with resins or gum-resins characterised by the presence of oxygen. The resins and gum-resins oxidise more readily than caoutchouc, the product of oxidation hardening and finally cracking. Hence the resins of caoutchouc are considered to be very injurious impurities in the material, and methods for estimating them are therefore very important.

Besides these resins, which are natural impurities, the consumer of caoutchouc often finds in it an artificial product known as factitious rubber or, more simply, as „substitute" („factice"). This body is prepared by boiling any of several oils with sulphur or sulphur chloride. It has a certain degree of elasticity, but does not possess the flexible character distinguishing true rubber; it will scarcely bear heating, and it accelerates the action of time on caoutchouc itself.

One of the most important processes in the manufacture of rubber goods is vulcanisation, an operation which has for its

object the combination of a certain proportion of sulphur with the caoutchouc under the influence of heat. If the temperature is too high during vulcanisation, substances of a resinous nature may be produced; if the amount of sulphur added is too large, or if it is not uniformly distributed throughout the mass of caoutchouc, the finished articles will contain free sulphur which will gradually combine with the caoutchouc, causing the goods to harden and eventually to crack. Hence a determination of the free sulphur becomes necessary.

One of the commonest falsifications of rubber consists in mixing new caoutchouc with old material that has been cast aside owing to incipient decomposition, i. e. material that is already more or less oxidised or resinified.

Therefore the four substances to which the investigator has to devote most attention, on account of the harm they do the material, are resins, substitutes, free sulphur, and old rubber. Grosser frauds are easily enough revealed. The author does not think that the presence of inorganic bodies can be considered as adulteration, because their existence in most rubber goods is as necessary as that of sand in mortar. Nevertheless, it is desirable to have a method of estimating them.

A separate determination of the deleterious substances mentioned is a long and difficult operation, especially if the chemist has no preliminary information in his possession, and if the mixture is very complicated, containing perhaps different gum-resins, different substitutes, free sulphur, old rubber, tar, asphaltum etc. In spite of the excellent work done by Henriques and Weber, our processes for the analysis of rubber goods require to be perfected. But although the analysis of products of unknown composition is difficult, it is easy to verify the composition of articles which should be made in accordance with a purchaser's instructions; the instructions being based on a knowledge of the properties of the material, on experience, and on the nature of the employment to which the articles are to be put, as is generally the case when the purchaser is a public authority. Moreover, the problem is facilitated by the fact that rubber articles always give the best results in actual use when the caoutchouc is free from any foreign substances except the mineral loading agents.

A consumer who is careful of his own interests will therefore invariably insist on having a rubber 1. composed essentially of pure Para caoutchouc, or containing less than a prescribed proportion of resin; 2. containing very little free sulphur; 3. free from substitutes; 4. containing no regenerated rubber.

* * *

In the author's opinion, the following analytical process gives sufficiently accurate results, and it may well serve as the model method into which the Sub-Committee will strive to introduce improvements and amplifications.

Determination of Resins. — A convenient quantity of the rubber is reduced to the finest possible powder by rubbing the sample with a rough file. If the rubber is pure or not loaded (i. e. if it floats), 1 gramme is prepared; if its flexibility shows that only a moderate amount of loading matter is present, 2 grammes are taken; but if it is so hard that it appears to be highly charged with mineral matter, 3 grammes of filings are made ready. The appropriate quantity of material is then brought into a 120 or 150 c. c. flask fitted with a reflux condenser, 50 c. c. of acetone are added, and the whole is gently boiled on a water bath for 4 hours. The mixture is thrown on a filter, and the residue is washed with acetone; the filtrate and washing being collected in a thin tared basin. The liquid in the basin is evaporated at a gentle heat, and the solid matter is weighed.

Determination of «Substitute». The substance remaining on the filter is introduced into a 120 or 150 c. c. flask connected with an inverted condenser as before, and boiled for 4 hours on a water bath with 50 c. c. of a 5 per cent solution of caustic soda in 95 per cent alcohol. The mixture is then thrown on a filter and the insoluble matter is washed with alcohol. To estimate the substitutes in the filtrate, 8 or 10 c. c. of water are added, and the whole of the alcohol is driven off, but the residue is not dried. It is next diluted with about 200 per cent of water, and dilute hydrochloric acid is introduced to render the liquid faintly acid. The fatty acids of the substitutes are thus precipitated, and, after standing for 12 hours, collect on the surface as flocks which

can easily be filtered off. After washing with hot water, the filter and its contents are laid on bibulous paper to absorb most of the water, and they are then transferred to a small Erlenmeyer flask containing about 20 cm^3 of ether. When all is dissolved, the ethereal liquid is filtered, the filtrate being received in a tared vessel. After driving off the ether at a gentle heat, a dry residue is obtained which represents the fatty acids of the substitutes; and, according to the author's experiments, its weight may be considered practically identical with the amount of substitute in the sample. It would, however, be interesting to ascertain whether some coefficient could not be deduced for correcting the weight obtained in this manner.

Determination of Sulphur. The following methods of estimating the sulphur that exists in different forms have been published by M. Pontis*), Chemist to the Under-secretary of the French "Postes et Télégraphes", and seem to the author to be worthy of recommendation.

1. Determination of Free Sulphur. Two grammes of rubber raspings are digested for six hours in 250% of boiling alcohol, and the residue is washed with alcohol. The filtrate is mixed with 20% of strong caustic soda solution, brought to the boil, and evaporated on a water bath. The residue is taken up in nitric acid diluted with half its volume of water, again boiled, and evaporated to dryness. The solid matter is dissolved in hydrochloric acid, and the sulphate in the liquid is precipitated with barium chloride in the usual manner.

2. Estimation of Sulphur combined with Substitutes. The material which has been freed from uncombined sulphur by means or substitute present. The insoluble portion is washed with boiling water, and the liquid is evaporated to dryness. It is taken up in dilute nitric acid, and further treated as in the preceeding paragraph,

3. Estimation of Total Sulphur. One gramme of the finely divided caoutchouc is triturated with a mixture of equal parts of pure manganese dioxide and sodium carbonate. A tall porcelain crucible is charged with one gramme of the oxidising mixture, the diluted rubber powder is next put in, and then another

*) Revue générale de Chimie pure et appliquée, January 10th, 1904.

layer of the oxidising mixture. The crucible is inserted into a muffle furnace, and kept at a red heat for an hour and a half. The mass is carefully treated with dilute hydrochloric acid, evaporated to dryness, dissolved again in boiling water containing a little hydrochloric acid, filtered if there is any residue, and the hot solution is precipitated with barium chloride as usual.

Determination of Inert Matter. A determination of the inert matter is important principally because it enables the analyst to ascertain the proportion of caoutchouc in the manufactured rubber, and so to calculate the proportion of resin in the caoutchouc. It is this proportion, indeed, that indicates most accurately the value of the material under examination.

If the rubber is loaded with fairly non-volatile matter, such as litharge, zinc oxide, talc, or chalk, the amount of inert matter present may be estimated by ignition. The sample is introduced into a porcelain crucible supported in a circular hole made in a sheet of asbestos millboard, the flame of a Bunsen burner being caused to play on the lower part of the crucible in such fashion that the caoutchouc distils away without igniting. This is easy to manage as the asbestos protects the interior of the crucible from all contact with the flame itself. When the caoutchouc is entirely carbonised, the power of the burner is increased, care being taken that the temperature does not exceed a dull red heat.

If the rubber is loaded with inert matter that decomposes easily, such as the sulphide of mercury or antimony, special processes for determining the proportion must be adopted, the selection of which is to form the subject of future investigation.

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